START

September 16, 1991

Meeting Minutes Transmittal/Approval
1100-EM-1 Operable Unit Managers Meeting
450 Hills St., Richland, Washington
August 14, 1991

FROM/APPROVAL: Robert K Stewart Date 9/18/91
Robert K. Stewart, 1100-EM-1 Operable Unit Manager (DOE-RL)

APPROVAL: David R. Einan Date 18 Sept 91
Dave Einan, 1100-EM-1 Unit Manager, EPA

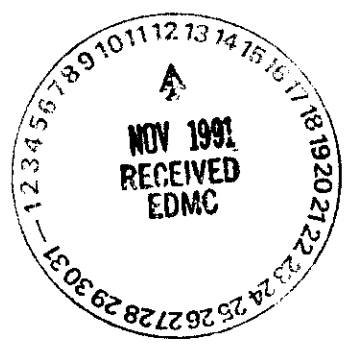
APPROVAL: Richard Hibbard Date 9/18/91
Richard Hibbard, 1100-EM-1 Unit Manager, WA Department of Ecology

Meeting Minutes are attached. Minutes are comprised of the following:

Attachment #1 - Meeting Summary/Summary of Commitments and Agreements
Attachment #2 - Attendance List
Attachment #3 - Agenda
Attachment #4 - Action Items Status List
Attachment #5 - Data Validation Procedures For Chemical Analyses
Attachment #6 - Radiochemical Analyses of Groundwater Monitoring Well Samples
Attachment #7 - DSI From S. Clark to J. Lerch re: Radiochemistry Analyses From K-25 Lab
Attachment #8 - Summary of Analytical Results for HRL B-4 & B-5 and Map of Sampling Locations
Attachment #9 - Summary of the Meeting on Geophysics Results and Proposed Test Pit Locations
Attachment #10 - Training Requirements for Hazardous Waste Site Workers
Attachment #11 - Incident Report - Unauthorized Disturbance of the Ephemeral Pool
Attachment #12 - WHC Memo - Improved Access Control to Inactive Waste Sites
Attachment #13 - Draft Revision to Milestones M-15-01B & M-15-01C

PREPARED BY: Kay Pratt Date 9/20/91
SWEC Support Services

CONCURRENCE BY: Dawn W. Cook Date 9/18/91
WHC RI Coordinator



1100-EM-1 Unit Managers Meeting
August 14, 1991

Distribution:

Chuck Cline, WDOE
Ward Staubitz, USGS
Mike Thompson, DOE-RL (A6-95)
Mary Harmon, DOE-HQ, (EM-442)

John Stewart, ACE
Linda Powers, WHC (B2-35)
Tom Wintczak, WHC (B2-15)
Mel Adams, WHC (H4-55)
Steven Clark, WHC (H4-55)
Brian Sprouse, WHC (H4-22)
Diane Clark, DOE-RL (A5-55)
Bill Price, WHC (S0-03)
Don Kane, Battelle EMO (K1-74)
Donna Lacombe, PRC
Jim Patterson, WHC
Michael Beavers, WHC (G1-66)
Earl Oxford, WHC (G4-11)

Ronald D. Izatt (A6-95)
Director, DOE-RL, ERD
June M. Hennig (A5-21)
DOE-RL, WMD
Roger D. Freeberg (A6-95)
Chief, Rstr. Br., DOE-RL,ERD
Steven H. Wisness
TPA Proj. Mgr.
Richard D. Wojtasek (B2-15)
Prgm. Mgr. WHC

Victor Wilde
Don Praast, GAO (A1-80)
KaeRae Parnell, WHC (H4-18)
Dave Einan, EPA (B5-01)
Michael Neely, PNL (K6-96)
Chuck Malody, ANF

ADMINISTRATIVE RECORD: 1100-EM-1; Care of Susan Wray, WHC (H4-51C)

Please contact Doug Fassett if there are any deletions or additions to this list.

Attachment #1

Meeting Summary and Summary of Commitments and Agreements
1100-EM-1 Unit Managers Meeting
August 14, 1991

Introduction

1. The draft WHC document Data Validation Procedures for Chemical Analyses was distributed to the regulators (see Attachment #5).

Work Progress

2. Wendell Greenwald (ACE) presented an update of the work progress (see Attachment #3). The groundwater analyses results from Weston (see Attachment #6) and technetium and thorium analyses (see Attachment #7) were provided. It is believed that technetium is the source of elevated *gross* beta measurements. Wendell Greenwald asked if it was worthwhile to perform special analyses for technetium. Technetium is very volatile and there was concern that some technetium might be lost during sample preparation or extraction of the sample (see Attachment #7). The options would be to accept less quantitative data or to spend more and have a longer turnaround time. Based upon the unvalidated data that will be provided to the regulators, a decision will be made on what action to take.

Action Item #11EM1.85: Information on cost, schedule, and other constraints associated with technetium analyses is to be provided to the regulators. The information is to be provided in a meeting on Wednesday, August 21. Action: Wendell Greenwald (8/14/91)

3. Wendell Greenwald presented sample results from Horn Rapids Landfill (HRL) (see Attachment #8). Also, Mr. Greenwald said that soil gas sampling at HRL would be done the week of August 12. It was agreed that geophysical work indicated anomalies that were equal to or greater than an equivalent ten drum anomaly. However, Joe Kunk (WHC) said there was probably not an anomaly greater than 50 drums.

Proposed Work

4. Wendell Greenwald presented an update of the proposed work (see Attachment #3). Geophysical work will be completed before the test pits are started. The anticipated date to begin the test pit work is September 3. Issues associated with this task include work in the 300 area process trenches which is competing for resources, and obtaining an agreement on the scope of work. ACE would like one more discussion on the number of test pits to be excavated (see Attachment #9 for the summary of a meeting on 7/24/91).

5. A letter from EPA Headquarters to the industrial hygienist *(Rich Silvey, WHC)* states what is necessary to achieve compliance during test pit excavation at the old asbestos burial pit. *The letter indicates that all excavated material would require containerization and disposal in a*

RCRA landfill. The industrial hygienist has been trying to find out which regulations must be complied with. Wendell Greenwald asked Dave Einan (EPA) to review the letter. Another issue is the possible need to notify the EPA Administrator in writing 45 days prior to beginning excavation. Dave Einan stated that he would check into this.

6. It is planned that eleven test pits be excavated. Bob Stewart (DOE) said that if the test pits were excavated to a depth greater than what was agreed to, the schedule for the excavations might need to be extended.

11EM1.86: EPA is to determine whether or not a notice is required 45 days before excavation begins on the test pits. The requirements for handling material excavated from the asbestos trench will also be determined. Action: Dave Einan (8/14/91)

7. Wendell Greenwald suggested that decontamination liquids related to test pit excavations be overpacked in a polydrum. Rich Hibbard (Ecology) questioned how overpack containers would be inspected, and how the overpack would prevent the liquid from freezing and would contain the frozen liquids. Concurrence was eventually reached allowing the overpacking of decontamination liquids. Bob Stewart (DOE-RL) asked if this agreement could be documented since there is no approved procedure. Mr. Stewart suggested that the procedure used at the 618-9 burial ground be followed if contamination was detected.

11EM1.87: ACE and WHC are to provide to the regulators the strategy for handling decon water related to excavation of the test pits. The strategy will also be attached to the September UMM minutes. Action: Wendell Greenwald (8/14/91)

8. ACE is proposing to sample test pits every five feet, and to perform field screening on every tenth backhoe bucket. X-Ray Fluorescence (XR-F) will be used as a field screening tool to determine if contaminants are above background. The contaminants of concern for the XR-F screening will be chromium, nickel, arsenic, and lead. Rich Hibbard said it must be determined in the field if XR-F is an applicable technique.

9. The disposition of Personal Protective Equipment at the test pits will be discussed in a meeting on August 15 at 9:30 AM. Investigation Derived Wastes will also be discussed.

10. Wendell Greenwald requested information on hazardous waste training from the regulators who will be entering the exclusion zones at the test pit excavations at HRL. Mr. Greenwald provided a form to obtain this information (see Attachment #10).

11. The Feasibility Study Phase I and Phase II will be available August 30th. The Supplemental Work Plan will be available September 30th if the Change Request is approved.

Advanced Nuclear Fuels Status

12. Advanced Nuclear Fuels (ANF) has changed its name to Siemens Nuclear Power (SNP). Doris Minor (SNP Support) presented an update on the status of SNP. A draft work plan is currently in internal discussion at SNP. SNP will be installing new wells but they will not be replacing the existing wells on a one to one basis. Bob Stewart (DOE-RL) asked when the work plan would be provided to DOE and the regulators. Doris Minor replied that the work plan would be available for distribution in late August or early September. Doris Minor said that SNP will take independent action until the regulators decide what is to be done at Horn Rapids Landfill. Data quality is going to be a highly valued objective in the work plan.

Ephemeral Pool Access Control

13. A road grader *disturbed soil* near the ephemeral pool during work on *the parking lot at the Vehicle Maintenance (1171) Building* on July 29 (see Attachment #11). However, the road crew did not encroach on any area that contained PCBs above the quantitation level. A risk assessment was run based on theoretical maximum PCB exposure to the workers. The risk was determined to be 4.34 X 10-9 risk. Signs have been put up at all *waste investigation* sites except for the 1171 antifreeze tank (due to operating constraints). WHC has developed some proposed changes to the procedures for access control at inactive waste sites (see Attachment #12).

Pesticide Spraying of HRL

14. On August 13 HRL was inadvertently sprayed with pesticides from a crop duster. The pesticides sprayed included Diathane, a fungicide, and Comite, a miticide.

"Good Faith" Dispute Status

15. An update on the "Good Faith" dispute was given. The DOE-RL dispute position was hand delivered to the two regulatory agencies on August 7.

Revision to Milestones Request Status

16. The draft Change Control Form for the revisions of milestones M-15-01B and M-15-01C was provided to the regulators (see Attachment #13).

Action Item Status - The status of the outstanding action items was presented (also see Attachment #4).

11EM1.55 Open; WHC will draft a letter to transmit the report.

11EM1.64 Open; a meeting will be scheduled after the groundwater summary report is received.

11EM1.65C Open; Dave Einan (EPA) is still working on the vinyl chloride issue.

11EM1.65D Open; it will be scheduled after the groundwater summary report is received.

11EM1.68 Closed; the geophysical meeting occurred on 7/25/91.

11EM1.72 Open

11EM1.73 Closed; an ecology RCRA inspector has not yet visited the site. Rich Hibbard (Ecology) stated that this action item could be closed.

11EM1.83 Closed; this action item is moot since Interim Response Measures will not be used.

Attachment #2

Attendance List

1100-EM-1 Unit Managers Meeting
August 14, 1991

Name	Organization	1100-EM-1 Responsibility	Phone
Harris, Allan	DOE-RL	Unit Manager	509-376-4339
Hibbard, Richard	Ecology	Unit Manager	206-493-9367
Cline, Chuck	Ecology	Geohydrology	206-438-7556
Einan, Dave	EPA	Unit Manager	509-373-3883
Greenwald, Wendell	ACE	Tech. Manager	509-386-9504
Liias, Raimo	ACE	Env. Engr.	509-522-6924
Baehre, Michael	ACE		509-376-1275
Staubitz, Ward	USGS	EPA Support	206-593-6510
Drost, Brian	USGS	EPA Support	206-593-6510
Clark, Steve	WHC	Env. Engr.	509-376-1513
Kunk, Joseph	WHC	Geophysics Support	509-376-4024
Mix, P.D.	WHC	Activities Engr.	509-373-2902
Knox, Kathy	CNES	GSSC, DOE-RL	509-376-5011
Fassett, Doug	SWEC	GSSC, DOE-RL	509-376-5011
Fryer, Bill	SWEC	GSSC, DOE-RL	509-376-9830
Shigley, Diane	SWEC	GSSC, DOE-RL	509-376-5038
McClung, Bill	SWEC	GSSC, DOE-RL	509-376-1853
Minor, Doris	ANF/SNP	Reg. Support	206-633-3208
Bower, Jay	ANF/SNP	Env. Support	206-869-6321

Attachment #3

Agenda

1100-EM-1 Unit Managers Meeting
August 14, 1991

1. Introduction

2. Work Progress
   - Groundwater Sampling
     - Quick turn-around sample results MW-21
     - Radiochemistry Data
       - 5th round groundwater sampling
       - Special analyses for technetium
   - Sample Results for HRL B-4 & B-5
   - Soil Gas Sampling

3. Proposed Work
   - Groundwater Sampling
   - Geophysical Surveys at HRL
   - Test Pits at HRL
     - Schedule
     - Number of test pits to be excavated
     - Final Sampling Plan and Scope of Work
       - Asbestos work
       - Over-pack liquids
       - Use of XR-F
       - Inorganics of concern
       - Disposition of PPE
     - Access to HRL during test pit excavation
   - Feasibility Study Phase I & II
   - Supplemental Work Plan

4. Advanced Nuclear Fuels Status

5. Ephemeral Pool Access Control

6. Pesticide Spraying of HRL

7. "Good Faith" Dispute Status

8. Revision to Milestone Request Status

9. Potential Dispute on Land Use and Risk Assessment Status

10. Action Item Status

Actions Items Status List

1100-EM-1 Operable Unit

Item No.	Action/Source of Action	Status
11EM1.55	WHC will review the Well Inventory Report to determine if the report is sufficient to send to the City of Richland and obtain an opinion from WHC Legal on the release. Action: Steve Clark (1/23/91, EM1-UMM)	Open. Draft a letter to transmit the report.
11EM1.64	Schedule a meeting with the City of Richland in mid-April to brief the city on the groundwater investigation and monitoring results, as they pertain to the city well field. ANF should be apprised of these activities. Action: Bob Stewart (DOE-RL), John Stewart (USACE), and Steve Clark (WHC) (3/20/91)	Open. Will be scheduled after ground water summary report received.
11EM1.65C	Dave Einan (EPA) will provide information regarding sampling and analysis for vinyl chloride, and investigate the handling of vinyl chloride issues on other EPA Region 10 sites. Action: Dave Einan (EPA) (3/1/91)	Open.
11EM1.65D	Contact appropriate DOE-RL and WHC personnel to investigate the possibility of having wells S37-E14, S40-E14, S41-E13A, S41-E13B and S43-E12 monitored under the site-wide monitoring program per section 2. Action Bob Stewart (DOE-RL) and Steve Clark (WHC) (3/1/91)	Open. Will be scheduled after ground water summary report received.

Item No.	Action/Source of Action	Status
11EM1.68	EPA and Ecology will schedule a meeting to review the Geophysical report and data, and notify DOE-RL and WHC so that representatives can attend. Action: Dave Einan (EPA) and Rich Hibbard (Ecology) (5/24/91).	Open. Meeting 7/25/91.
11EM1.71	Bob Stewart will attempt to get the radionuclide analyses from the laboratories. Action: Bob Stewart (5/24/91).	Closed.
11EM1.72	Investigate use of the C-018 Water Treatment Facility to treat contaminated groundwater from the HRL plume. Action: Bob Stewart (5/24/91) - Wendell Greenwald (6/20/91).	Open.
11EM1.73	Investigate the red drum sitting near the burn cage at HRL. Action: Steve Clark (WHC) (6/6/91).	Open. 6/13/91
11EM1.75	Locate and collect IRM and ROD guidance documents and copies of the INEL IRM documents. Action: Bob Stewart (DOE-RL), Dave Einan (EPA), Raimo Liias (USACE) (6/6/91).	Closed.
11EM1.76	The USACE will construct a flow diagram of the process for doing the IRM's and leading to the final ROD. Action: Wendell Greenwald (USACE) and John Stewart (USACE) (6/6/91).	Closed. Presented 6/20/91 Revisions Presented 7/17/91.

Item No.	Action/Source of Action	Status
11EM1.78	Provide Rich Hibbard (Ecology) with separate copies of the change request submittals to insure his receipt of same. Action: Bob Stewart (DOE-RL) (6/6/91).	Closed.
11EM1.79	EPA will evaluate the proposal to use MTCA's soil cleanup levels in lieu of performing a risk assessment for each IRM, recognizing that MTCA is an ARAR. Action: Dave Einan (EPA) (6/6/91).	Closed.
11EM1.80	EPA and Ecology are to review the sample plan for the characterization of the waste from the excavation of the test pits. Comments are to be provided by July 3. The comments will include results of research by EPA and Ecology on the handling of investigation derived waste at other landfills. Action: Dave Einan and Rich Hibbard (6/20/91)	Closed.
11EM1.81	Comments on the outline for the proposed plan for IRMs were requested by July 3 by DOE. Action: Bob Stewart, Rich Hibbard and Dave Einan (6/20/91)	Closed.
11EM1.82	EPA and Ecology are to discuss the MTCA based soil cleanup levels, revise the Soil Cleanup Level table, provide the revised table to DOE and provide conclusions on factors that control the required cleanup levels. This information is to be used for a discussion on adding additional "soil sites" to the group of proposed soil remediation IRMs. Action: Dave Einan and Rich Hibbard (6/20/91)	Closed.

Item No.	Action/Source of Action	Status
11EM1.83	Donna Lacombe will provide a copy of the Idaho (Idaho National Engineering Loboratories) interim response measure (IRM) procedure to DOE. Action: Donna Lacombe (7/17/91).	Open.

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Draft 7/91

Submitted to:

Westinghouse Hanford Company

Draft

Data Validation Procedures for Chemical Analyses

DRAFT
DATA VALIDATION PROCEDURES
FOR
CHEMICAL ANALYSES

July 26, 1991

903-1248

CONTENTS

CONTENTS (Cont.)

CONTENTS (Cont.)

CONTENTS (Cont.)

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CONTENTS (Cont.)

LIST OF TABLES

LIST OF APPENDICES

LIST OF ACRONYMS

%D	percent difference
AA	Atomic absorption spectrophotometry
BFB	bromofluorobenzene
CCV	continuing calibration verification
CLP	Contract Laboratory Program
CN	cyanide
COD	Chemical Oxygen Demand
CRQL	Contract Required Quantitation Limit
CVAA	cold vapor atomic absorption spectrophotometry
DBC	dibutylchlorendate
DFTPP	decafluorotriphenylphosphine
DOE	United States Department of Energy
EPA	U. S. Environmental Protection Agency
FLAA	flame atomic absorption spectrophotometry
GC/MS	gas chromatography/mass spectrometry
GC	gas chromatography
GFAA	graphite furnace atomic absorption spectrophotometry
GPC	gel permeation chromatography
HCL	hydrochloric acid
HEIS	Hanford Environmental Information System
EDMC	Environmental Data Management Center
EII	Environmental Investigation Instruction
h	hour
HG	mercury
HNO3	nitric acid
HRGC	high resolution gas chromatography
HRMS	high resolution mass spectrometry
IC	ion chromatography
ICP	Inductively coupled plasma emission spectrometry
ICS	ICP interference check sample
ICV	Initial calibration verification
IDL	Instrument detection limit
ISD	ICP serial dilution
MDL	Method detection limit
MSA	Method of standard additions
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NIST	National Institute of Standards and Technology
PCB	polychlorinated biphenyl
PCDD	polychlorinated dibenzo-p-dioxin
PCDF	polychlorinated dibenzofuran
PCDPE	polychlorinated diphenyl ether

LIST OF ACRONYMS (Cont.)

PFK	perfluorokerosene
QA	quality assurance
QAPjP	quality assurance project plan
QC	quality control
RF	response factor
RIC	reconstructed ion chromatogram
RPD	relative percent difference
RRF	relative response factor
RSD	relative standard deviation
RT	retention time
SDG	sample delivery group
SOW	statement of work
TAL	target analyte list
TCL	target compound list
TIC	tentatively identified compound
TOC	Total Organic Carbon
TOX	Total Organic Halogen
TWP	technical work plan
VOC	volatile organic compound
WHC	Westinghouse Hanford Company

1.0 INTRODUCTION

This statement of work is intended to provide guidance to Westinghouse Hanford Company (WHC) subcontractors tasked with the validation of chemical analytical data produced as a result of Hanford Site environmental investigations. The procedures described herein are applicable to the review and validation of investigation data, remediation data and verification data resulting from environmental investigations. Validation is a Quality Assurance (QA) review process, and is defined as the process of reviewing a body of analytical data against the criteria established herein in order to ensure that the data are acceptable for their intended use. This document shall be appended to all procurement documents for validation services; it is intended for use by trained chemists or scientists in conjunction with the applicable project-specific work plans, QA Project Plans (QAPjPs) analytical method references, and laboratory statements of work during the review of the following chemical analytical data:

- EPA Contract Laboratory Program (CLP) Target Compound List (TCL) organic compounds;
- CLP Target Analyte List (TAL) inorganic analytes;
- Chlorinated herbicides; 2,4-D and 2,4,5-TP (Silvex);
- Polychlorinated dibenzo(p)dioxins and dibenzofurans; and
- Miscellaneous compounds, analytes and parameters determined by wet chemical methods (see Section 9).

The procedures contained herein are not intended to be exclusive for the above analytical methods but may be applied to other procedures. Where applicable, alternate analytical methods (non-CLP) are referenced as related to the validation process.

1.1 GENERAL REQUIREMENTS

The WHC project coordinator will provide the data validation subcontractor current copies of the applicable project specific work plans, sampling plans, QAPjPs, laboratory statements of work, laboratory QAPjPs, and laboratory standard operating procedures (SOPs), specifying the analytes of interest, reference analytical methods, contract required quantitation limits (CRQLs), and goals for analytical precision, accuracy, and completeness.

Sections 2 through 10 of this document provide the necessary guidance for the performance of specific categories of data validation reviews. During data validation, the reviewer will be required to complete validation checklists and summary forms for

documentation and reporting purposes. Appendices A and B provide copies of data review checklists and summary forms that are to be completed.

The data reviewers shall complete several tasks on a sample delivery group basis during validation and review of the laboratory data packages. A sample delivery group shall be defined as a group of samples (usually 20 or fewer) reported within the same laboratory data package. These tasks are summarized as follows:

- Receipt of the data package, date stamping the data package and making duplicate copies of the sample concentration reports or report forms;
- Organize and review the data package for completeness as described in Sections 3 through 9 below, and document completeness of the data package on the applicable data validation checklist;
- Review and validate the data package according to the procedures described in Sections 3 through 10 and document the review using the checklists and forms described in Section 2. Data that are rejected shall be eliminated from any further reivew or consideration.
- Check the result calculations at the frequency specified in Section 2.4.
- Resolve discrepancies in the data package discovered as a result of the review with the laboratory by telephone contact and document all contacts on the appropriate forms described in Section 2;
- Following completion of data validation, prepare a narrative summary of the data acceptability and prepare a summary of the validated results in tabular and electronic format using the guidelines specified in Section 10; and
- Submit the data validation report including the narrative summary, electronic data, checklists, summary forms, and copies of the as-received laboratory sample concentration reports to the WHC project manager within 21 days after receipt of the data package from WHC or the contract laboratory. The original laboratory data packages shall be maintained for a period no longer than three months after the date of receipt by the reviewer, after which the packages shall be returned to the WHC project manager.

2.0 SPECIFIC REQUIREMENTS

This section presents specific requirements that apply to all data review activities specified in this statement of work.

2.1 RECORDS MANAGEMENT

The data reviewer(s) shall have a records management and document control program established that meets the following general requirements. Upon receipt of the data package by the data reviewer, the data package shall be date stamped and a duplicate record of the sample concentration reports shall be made for use during the data validation and for transmittal in the final validation report. The original laboratory data packages will be held by the data reviewer for a period no longer than three months after the date of receipt by the data reviewer, after which the packages shall be returned to the WHC project manager.

2.2 DATA PACKAGE COMPLETENESS

After receipt of the data package, the data validator shall organize and review the data package for completeness by checking for the deliverable items listed in the appropriate data validation checklist (see Section 2.4 and Appendix A) using the guidelines specified in sections 3 through 9. Observation of omitted deliverable items shall prompt the reviewer to contact the laboratory by telephone to resolve discrepancies and request the necessary omitted deliverables. All telephone conversations with the laboratory must be documented on the appropriate form (see Section 2.4).

2.3 TECHNICAL AND MANAGEMENT REQUIREMENTS

Contractors shall have an organization with defined responsibilities and defined technical capabilities for individuals responsible for successful completion of data validation reviews. The contractor shall designate personnel to conduct the following tasks for all WHC data review contracts or task orders.

- Data Validators - Data validators shall be responsible for conduct of data validation, and reporting activities as assigned by the contractor project manager. Data validators shall have a minimum of a bachelor's degree in chemistry or any physical or life science with a minimum of one year of experience in laboratory analysis or data validation.

- Project Manager - Project managers shall be responsible for overall management and direction of the data validation, and reporting activities and assignment of responsibilities to validation personnel. Project managers shall have a minimum of a bachelor's degree in chemistry, physical or life science with a minimum of three years experience in laboratory analysis or data validation and including at least one year of supervisory experience.

- Document Custodian - Document custodians shall be responsible for records management activities associated with data validation as assigned by the project manager. Document custodians shall have a minimum of a bachelor's degree in any field and one year experience in records management.

- Quality Assurance Officer - Quality assurance officers shall be responsible for verification of compliance with the data validation procedures embodied in this scope of work. Quality assurance officers shall have a minimum of a bachelor's degree in any field and one year experience in laboratory analyses or data validation, and shall have sufficient independence from project management, cost and schedule concerns to permit the identification and resolution of quality problems related to the validation process.

2.4 DATA VALIDATION AND REPORTING

Data validation contractors shall conduct the data validation using the procedures and criteria specified in Sections 3 through 9. Calculation checks conducted during validation shall be performed at the following frequencies for each category of site data.

- Investigation data -- All reported laboratory results for at least 20 percent of the samples contained in the SDG and 100 percent of the reported quality control samples (duplicates, matrix spikes, field blanks and performance audit samples) shall be calculated and verified against the raw data. If possible, at least one-half of the samples selected for recalculation must contain positive results for the target compounds analyzed.

- Remediation data -- All reported laboratory results for at least 10 percent of the samples contained in the SDG and 100 percent of the reported quality control samples (duplicates, matrix spikes, field blanks and performance audit samples) shall be recalculated and verified against the raw data. If possible, at least one-half of the samples selected for recalculation must contain positive results for the target compounds analyzed.

- Verification data -- All reported laboratory results for 100 percent of the samples contained in the SDG and 100 percent of the reported quality control samples

(duplicates, matrix spikes, field blanks and performance audit samples) shall be recalculated and verified against the raw data.

Unless otherwise specified in the appropriate sections of the data review requirements calculation checks performed on all other reported data shall be conducted at a frequency of five percent.

All validation activities shall be documented using the checklists and forms described in Sections 2.4.1, 2.4.2 and Appendices A and B. In addition following completion of the validation checklists and forms, a report shall be prepared that summarizes the validation conducted using the guidance provided in Section 10.

2.4.1 Data Validation Checklists

Each data validation checklist described below consists of a question and answer form to be completed for the data review, the action required in the case of unacceptable data, and space for comments for the reviewer to include additional information as necessary to explain the review.

2.4.1.1 Volatile Organic Data Review Checklist - Form A-1

This checklist is to be completed during review of volatile organic analysis data packages using the criteria specified in Section 3.

2.4.1.2 Semivolatile Organic Data Review Checklist - Form A-2

This checklist is completed during review of semivolatile organic analysis data packages using the criteria specified in Section 4.

2.4.1.3 Pesticide/PCB Data Review Checklist - Form A-3

This checklist is completed during review of pesticide/PCB organic analysis data packages using the criteria specified in Section 5.

2.4.1.4 Herbicide Data Review Checklist - Form A-4

This checklist is completed during review and validation of herbicide organic analysis data packages using the criteria specified in Section 6.

2.4.1.5 Dioxin/Furan Data Review Checklist - Form A-5

This checklist is completed during review and validation of dioxin/furan organic analysis data packages using the criteria specified in Section 7.

2.4.1.6 Inorganic Data Review Checklist - Form A-6

This checklist is completed during review and validation of inorganic analysis data packages using the criteria specified in Section 8.

2.4.1.7 Wet Chemistry Data Review Checklist - Form A-7

This checklist is completed during review and validation of wet chemistry data packages using the criteria specified in Section 9.

2.4.2 Data Validation Summary Forms

The following sections provide a brief description of the data validation summary forms (Appendix B) that are to be completed by the reviewer and attached to the validation checklist for inclusion in the validation report.

2.4.2.1 Holding Time Summary - Form B-1

This form is to be completed for each sample group documenting the dates of sample collection, preparation, and analysis.

2.4.2.2 Calibration Data Summary - Form B-2

This form is to be completed for each sample group documenting the compounds that exceed the calibration criteria and the affected samples.

2.4.2.3 Blank and Sample Data Summary - Form B-3

This form is to be completed for each sample group documenting the compounds or analytes detected in all blanks and samples.

2.4.2.4 Accuracy Data Summary - Form B-4

This form is to be completed for each sample group during review of matrix spikes, surrogates, control samples, and performance audit samples documenting the compounds or analytes that exceed the specified criteria and the affected samples.

2.4.2.5 Precision Data Summary - Form B-5

This form is to be completed for each sample group during review of MS/MSD, field duplicates, laboratory duplicates and split samples documenting the compounds or analytes that exceed the specified validation criteria and the affected samples.

2.4.2.6 Calculation Summary - Form B-6

This form is to be completed whenever reported data are recalculated and verified.

2.4.2.7 Data Qualification Summary - Form B-7

This form is to be completed at the conclusion of the data review documenting all qualifications necessary as a result of the data review.

2.4.2.8 Contact Summary - Form B-8

This form is to be completed whenever a telephone or other contact is made with the laboratory or other personnel that relate to the review of the subject analytical data.

2.5 Data Reporting Qualifiers

Data reporting qualifiers to be applied as a result of data validation are summarized below:

U - Indicates the compound or analyte was analyzed for and not detected. The value reported is the sample quantitation limit corrected for sample dilution and moisture content by the laboratory.

UJ - Indicates the compound or analyte was analyzed for and not detected. Due to identified quality control deficiency identified during data validation the value reported may not accurately reflect the sample quantitation limit.

J - Indicates the compound or analyte was analyzed for and detected. The associated value is estimated but the data are usable for decision making processes.

R - Indicates the compound or analyte was analyzed for and due to an identified quality control deficiency the data are unusable.

NJ - Indicates presumptive evidence of a compound at an estimated value.

N - Indicates presumptive evidence of a compound.

3.0 VOLATILE ORGANIC DATA REVIEW REQUIREMENTS

This section presents data review requirements for volatile organic analysis conducted using the CLP Statement of Work (SOW), (EPA 1988a). The method of analysis is based on a modification of Method 624 from 40 CFR Part 136 to analyze organic compounds listed on the CLP target compound list (TCL). The data review requirements described herein may be applied to the review of data produced using Method 8240 or Method 8260 (EPA 1986).

3.1 DATA PACKAGE COMPLETENESS

After receipt of the data package and completion of records management activities detailed in section 2, the reviewer shall organize the data package according to the order of deliverables specified in section 1 of the data validation checklist (Appendix A, Form A-1). Missing data review items that the reviewer deems necessary for completion of the validation shall require the reviewer to contact the laboratory for submittal of the needed item. All contacts with the laboratory must be documented on the appropriate form (Appendix B, Form B-8).

3.2 HOLDING TIMES

Review the chain-of-custody forms, laboratory reports, and quantitation sheets for date of sample collection and analysis. All samples must be collected in the proper containers protected from light, shipped under chain-of-custody and stored at 4°C until the time of analysis. Analysis must be completed within 7 days of collection for unpreserved water samples and 14 days of collection for preserved water samples and chilled soil samples. Calculate holding times as follows: holding time, days = (sample analysis date - sample collection date). Complete the holding time summary form (Appendix B) and if the criteria were not met, qualify all associated data as estimated (J for detects, UJ for non-detects).

3.3 INSTRUMENT CALIBRATION AND TUNING

Review the laboratory reports, quantitation sheets, and raw data to verify the laboratory has tuned and calibrated the instrument prior to sample analysis and periodically through the sample analysis batch. Criteria for the review of tuning and calibration data are provided in Sections 3.3.1 through 3.3.3.

3.3.1 GC/MS Tuning

Review the tuning reports, mass listings and spectra and verify the laboratory has conducted an acceptable tune using bromofluorobenzene (BFB) for every 12th time period in which samples were analyzed. Using the tuning criteria listed in Table 3-1, check for calculation and transcription errors and the proper reporting of significant figures. The proper significant figures are listed in the ion abundance criteria column of Table 3-1. Check for errors in calculation of percent abundance values using the mass abundance values reported in the raw data. For example, calculate the percent abundance for m/z 96 relative to m/z 95 using the following formula: % abundance = (relative abundance of m/z 96 / relative abundance of m/z 95) x 100. If the ion abundance criteria are not met, check that the ion ratios for m/z 95/96, 174/175, 174/176, and 176/177 are within specification and check that the expanded criteria for m/z 50 and m/z 75 are met. Expanded criteria for m/z 50 are 11.0 - 50.0% of m/z 95. Expanded criteria for m/z 75 are 22.0 - 75.0% of m/z 95. If the tuning criteria are not met but the tuning is within the expanded criteria qualify associated data as estimated (J for detects, UJ for non-detects). If all tuning criteria are missed, qualify all associated data as unusable (R).

3.3.2 Initial Calibration

Review the calibration forms, quantitation reports and chromatograms to verify the laboratory has conducted an acceptable five-point calibration in accordance with the CLP CLP SOW prior to sample analysis. Verify that all relative response factor (RRF) values are ≥0.05, all relative standard deviation (RSD) values are ≤30% and check for calculation errors. Recalculate at least 20% of the individual and average RRF values and RSD values using the following formulas:

- RRF = $(A_x \times C_{is}) / (A_{is} \times C_x)$ where,

  A_x = the area of the characteristic ion for the compound.
  A_{is} = the area of the characteristic ion for the specified internal standard (see EPA 1988a).
  C_x = the concentration of the compound (ng/ul).
  C_{is} = the concentration of the specified internal standard (see EPA 1988a).

- Average RRF = (Sum of individual RRF values) / number of individual RRF values (usually 5).

- RSD = (Standard Deviation of the individual RRF values) / (Mean of the individual RRF values).

Table 3-1. Volatile Organic GC/MS Tuning Criteria

MASS	ION ABUNDANCE CRITERIA
50	15.0 - 40.0% of mass 95
75	30.0 - 60.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0 - 9.0% of mass 95
173	Less than 2.0% of mass 174
174	Greater than 50.0% of mass 95
175	5.0 - 9.0% of mass 174
176	Greater than 95.0%, but less than 101.0% of mass 174
177	5.0 - 9.0% of mass 176

(Source: EPA 1988a)

Complete the calibration data summary form (Appendix B) noting compounds that exceed the calibration criteria and the affected samples. If any RRF value is out of specification qualify all detected results as estimated (J) and all non-detects as unusable (R). If any RSD value is out of specification qualify all associated data as estimated (J for detects, UJ for non-detects).

3.3.3 Continuing Calibration

Review the continuing calibration forms, quantitation reports and chromatograms to verify the laboratory has conducted an acceptable calibration check in accordance with the CLP CLP SOW for every 12h analytical period in which samples were analyzed. Verify that all RRF values are ≥0.05, all %D values are ≤25%, and check for calculation errors. Recalculate at least 20% of the individual RRF and %D values using the following formulas:

- RRF = (see section 3.3.2)
- $\%D = [(RRF_i - RRF_c) / RRF_i] \times 100$ where,

  RRF_i = the average RRF from the initial calibration.
  RRF_c = the RRF value from the continuing calibration.

Complete the calibration data summary form (Appendix B) noting the compounds that exceed the criteria. If any RRF value is out of specification qualify all associated detected results as estimated (J) and all non-detects as unusable (R). If any other %D value is out of specification qualify all associated data as estimated (J for detects, UJ for non-detects).

3.4 BLANKS

Blanks are analyzed as a means of determining contamination introduced by the laboratory or sampling operations. Contamination may be introduced from the sample handling, sample processing, sample containers, sampling procedures and sample equipment.

3.4.1 Laboratory Blanks

Review the report forms, quantitation reports and chromatograms and verify the laboratory has conducted an acceptable method blank analysis on all matrices for every 12h time period in which samples were analyzed. The method blank analysis must be conducted after the calibration standard analyses and may contain less than or equal to five times the Contract Required Quantitation Limit (CRQL) of the following common laboratory contaminants; methylene chloride, acetone, toluene, and 2-butanone. Complete

the blank data summary form (Appendix B) noting the compounds that are detected in the laboratory blanks and affected samples.

Positive sample results for common laboratory contaminants less than 10 times the blank concentration (less than 5 times for other contaminants) are qualified by elevating the quantitation limit according to the following criteria:

- If the sample result is greater than the Contract Required Quantitation Limit (CRQL), but is less than five or ten times the associated highest blank result, the result is qualified as non-detected (U) at the reported concentration.

- If the sample result is less than the CRQL and is less than five or ten times the associated highest blank result, the result value is raised to the CRQL level and qualified as non-detected (U).

- If the sample result is greater than the CRQL and greater than five or ten times the associated highest blank result, no qualification is necessary.

Unidentified tentatively identified compounds (TIC) present in the samples and blanks with retention times that are within one minute of each other must be considered non-detects (U) if the sample concentration is less than five times the highest concentration in any blank.

3.4.2 Field Blanks

Following the review of laboratory blank data, review the chain of custody and sample analysis request documentation to identify the field blanks. If necessary contact the WHC project coordinator to obtain the required information.

3.4.1.1 Equipment Blanks

Review the report forms, quantitation reports and chromatograms. If positive results are reported, it may indicate that decontamination procedures were inadequate in the sampling process, or inherent to the equipment used and this should be noted in validation report narrative. Complete the blank data summary form (Appendix B) noting the compounds that are detected in the equipment blanks and the affected samples. Qualify any associated data as non-detected (U) for all positive results that are less than five times the highest valid field blank result.

3.4.1.2 Trip Blanks

Review the field sampling documentation, if necessary to identify the trip blanks. Review the report forms, quantitation reports and chromatograms and complete the blank data summary form (Appendix B) noting the compounds that are detected in the trip blanks. Qualify any associated data as non-detected (U) for all positive sample results that are less than five times the highest valid trip blank result.

3.5 ACCURACY

Laboratory performance and compliance with project specific and analytical accuracy requirements is determined by a review of surrogate and matrix spike results. The laboratory should conduct at least one matrix spike and matrix spike duplicate analysis for each sample delivery group (SDG) and all samples and blanks shall be spiked with surrogate compounds. A list of the surrogate and matrix spiking compounds and concentrations are found in the CLP SOW, (EPA 1988a).

3.5.1 Surrogate Recovery

Review the surrogate summary forms, quantitation reports and chromatograms, check all surrogate results for calculation errors and verify that recoveries are within the specifications outlined in the CLP SOW (EPA 1988a). Recalculate surrogate recoveries using the following formula: $\%R = (Q_d/Q_a) \times 100$ where, %R = percent recovery, Q_d = quantity of surrogate determined from the analysis, and Q_a = the quantity of surrogate added to the sample. If calculation errors are noted contact the laboratory for clarification and submittal of correct data if necessary. Complete the accuracy data summary form (Appendix B) for surrogates that are out of specification and qualify all associated sample results as estimated (J for detects, UJ for non-detects) for surrogates out of specification but greater than 10% recovery. Qualify all associated detected results as estimated (J) and non-detects as unusable (R) for surrogate recoveries less than 10%. If method blank surrogates are out of specification and associated sample surrogates are acceptable no qualification is necessary, however, the laboratory should be contacted for an explanation. If method blank and associated sample surrogates are out of specification, contact the laboratory for an explanation.

3.5.2 Matrix Spike Recovery

Review the matrix spike summary forms, quantitation reports and chromatograms, check for calculation errors and verify the laboratory has conducted at least one Matrix Spike/Matrix Spike Duplicate (MS/MSD) analysis for each sample group using the requirements specified in the CLP SOW (EPA, 1988a). Recalculate all MS/MSD recoveries using the following formula:

- MS percent recovery = [(SSR - SR) / SA] x 100 where,

  SSR = spiked sample result,
  SR = sample result, and
  SA = spike concentration added from the spiking mixture.

Complete the accuracy data summary form (Appendix B) for MS/MSD compounds that are out of specification. Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the results in the validation narrative. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

3.5.3 Performance Audit Samples

Contact the WHC project coordinator to obtain information regarding the identity and composition of any performance audit samples submitted with the sample batch. Review the report forms, quantitation reports and chromatograms, complete the accuracy data summary form (Appendix B), calculate the percent recovery of each spiked compound and compare to the quality control limits specified by the supplier of the sample. If the sample results are outside the control limits, advise the WHC project coordinator, contact the laboratory for an explanation, and discuss in the validation narrative.

3.6 PRECISION

The precision of the overall measurement system is determined by the reproducibility of MS/MSD analyses, field duplicates and split samples.

3.6.1 Matrix Spike/Matrix Spike Duplicate Samples

Review the MS/MSD reports, quantitation reports and chromatograms, verify the laboratory has conducted at least one MS/MSD analysis for the sample delivery group and check for calculation errors. Recalculate all MS/MSD RPD values using the following formula:

- MS/MSD RPD = (D1 - D2) / [(D1 + D2) / 2] x 100 where,

  RPD = relative percent difference,
  D1 = MS value, and
  D2 = MSD value (duplicate).

Complete the precision data summary form (Appendix B) for compounds that exceed the RPD criteria listed in the CLP SOW (EPA, 1988a). Review the MS/MSD results in conjunction with other QC data such as field duplicates and note the results in the validation narrative. If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

3.6.2 Field Duplicate Samples

Review the chain of custody and sample analyses request documentation to identify field duplicates and their corresponding samples, report forms, quantitation reports, and chromatograms. Calculate the RPD of the positive results for each compound using the CRQL if one of the results is a non-detect. Since precision criteria have not been developed by EPA, the inorganic analysis duplicate criteria have been applied to the evaluation of organic analysis field duplicate results. RPD limits for field duplicates (where both results are greater than five times the CRQL) are 20% for waters and 35% for soils. When one or both results are less than five times the CRQL the limits are ±CRQL for waters and ±2xCRQL for soils. Complete the precision data summary form (Appendix B) and note the results of the field duplicate analyses in the validation narrative.

3.6.3 Field Split Samples

Review the chain of custody and sample analysis request documentation to identify field split samples and the corresponding report forms, quantitation reports and chromatograms. Contact the WHC project coordinator to obtain information about the sample results from participating referee laboratories. Refer to section 3.6.2 for review and reporting requirements. Complete the precision data summary form (Appendix B) and note the results of the field split analyses in the validation narrative.

3.7 SYSTEM PERFORMANCE

Review the report forms, chromatograms and quantitation reports for evidence of RIC baseline anomalies, retention time shifts, extraneous peaks, low resolution and peak anomalies. Check that positive results are not affected by abrupt changes in baseline caused by leaks in the MS system or GC column bleed. In addition, look for positive results affected by coeluting compounds and ensure that the detected compound is resolved by at least ≥25%. If in the reviewer's informed professional judgement that quantitative sample results may be biased due to system performance anomalies such judgement must be addressed in the validation narrative and the affected results shall be qualified accordingly.

3.7.1 Internal Standards Performance

Review the report forms, quantitation reports and chromatograms, verify that area counts and retention times comply with CLP SOW (EPA, 1988a) requirements and check for calculation and transcription errors. If area counts for a particular internal standard are outside the +100 to -50 percent limits qualify associated sample results as estimated (J for detects, UJ for non-detects). If area counts are outside the limits and relative retention time criteria are > $\pm$ 30 seconds, qualify associated non-detects as unusable (R).

3.8 COMPOUND IDENTIFICATION AND QUANTITATION

Review the quantitation reports, chromatograms, and spectra for laboratory standards, samples and blanks to verify positive identification of TCL compounds. Verify that retention times for positive results in samples are within ±0.06 relative retention time units of the associated calibration standard, and review chromatograms to verify that all peaks are identified. Compare sample mass spectra to laboratory-generated standard spectra for compliance with the following criteria:

- All ions present in the standard at a relative intensity of 10% or greater are present in the sample spectrum,
- Relative intensities of standard and sample spectra agree within 20%, and
- Ions greater than 10% in the sample spectrum and not in the standard are identified and explained.

Qualify all affected positive results as follows:

- If the retention time criteria and mass spectral criteria are exceeded, qualify the results as unusable (R) and note in the validation narrative.
- If the reviewer determines that incorrect identifications were made as a result of cross-contamination between analyses then affected data should be qualified as unusable (R) and noted in the validation narrative.

3.8.1 Reported Results and Quantitation Limits

Review the report forms, quantitation reports and chromatograms, verify that correct internal standards, quantitation ions, and RRF values were used for quantitation and check for calculation errors. In addition, review sample dilutions and dry weight factors to verify accurate adjustments for CRQL values. Recalculate at least 20% of all results using the following formula:

- Water samples, $\mu g/L = (A_x \times I_s) / (A_{is} \times RRF \times V_o)$ where,

  A_x = area of the quantitation ion for the compound,
  A_{is} = area of the quantitation ion for the specified internal standard,
  RRF = relative response factor for the compound from the daily standard,
  I_s = amount of the specified internal standard in nanograms (ng), and
  V_o = the volume of water purged in milliliters taking into account any dilutions used by the laboratory.

- Soil samples, medium level, $\mu g/Kg = (A_x \times I_s \times V_t) / (A_{is} \times RRF \times V_i \times W_s \times D)$ where,

  A_x, A_{is}, I_s, RRF = same as for water samples, above,
  V_t = the volume of the medium level extract in microliters (ul),
  V_i = the volume of the extract added (ul) for purging,
  D = the percent solids, and
  W_s = the weight of sample extracted or purged

- Soil samples, low level, $\mu g/Kg = (A_x \times I_s) / (A_{is} \times RRF \times W_s \times D)$

  A_x, I_s, A_{is}, RRF, W_s, D = same as for medium level soils, above.

Determine if the laboratory was able to meet the project specific CRQL goals. In the absence of known or suspected analytical interferences, if the laboratory was unable to determine any compound within five times the CRQL, the laboratory must be contacted for clarification. Clarifications so obtained must be addressed in the validation narrative.

3.8.2 Tentatively Identified Compounds (TIC)

Chromatographic peaks may be present in an analysis that are not TCL analytes, surrogates, or internal standards and are considered tentatively identified compounds (TIC) and must be qualitatively identified by the laboratory.

Verify that spectral library searches were conducted for the ten largest unknown peaks in accordance with the CLP SOW (EPA 1988a). Qualify as non-detects (U) all compounds including common laboratory contaminants if present in the blanks using the blank review criteria in Section 3.4. A list of common laboratory contaminants often detected and reported as TIC are presented in Table 3-2.

Table 3-2. Common Laboratory Contaminants Identified as TIC Compounds

Carbon dioxide
Siloxane compounds (common GC column bleed artifacts)
Diethyl ether
Hexane
Freon compounds (Freon 112, Freon 113)
Phthalate compounds less than 100 µg/L (water samples) and less than 4,000 µg/Kg (soil samples)
Solvent preservatives (cyclohexene and by-products)
Reaction products of acetone (diacetone alcohol)

(Source: EPA 1988a)

In addition, the assessment of blanks should include an examination for TIC peaks present but not reported in the blanks due to the peaks being less than 10% of the associated internal standard height. All TIC peaks should be evaluated against the following criteria and considerations:

- Major ions (> 10% relative intensity) in the reference spectra should be present in the sample spectra;
- Relative intensities of major ions in the sample spectra should be within ±20% of the associated reference spectra;
- Molecular ions in the reference spectra should be present in the sample spectra;
- Ions present in the sample but not in the reference spectra should be reviewed for the possibility of interferences caused by co-elution of other TIC or possibly TCL compounds;
- If the TIC is not found in the blanks but is a suspected laboratory contaminant the result should be qualified as unusable (R);
- If the library search reveals more than one acceptable compound match the result may be qualified as a non-specific isomer; and
- If the sample(s) contain groups of TIC results that are similar isomers the reviewer should summarize and report all similar isomers as total, such as all alkanes reported as total alkanes or all unknowns reported as total unknowns.

If the reviewer determines that TIC identification is in error the associated sample results should be qualified as non-detects (U) or unusable (R). If the TIC identification is determined to be valid, the results are to be qualified as presumptive and estimated (JN).

3.9 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A), the applicable data summary forms (Appendix B), and briefly summarize any technical problems associated with the data. Summarize the overall quality and useability of the data in the validation narrative according to the requirements of Section 10.

4.0 SEMIVOLATILE ORGANIC DATA REVIEW REQUIREMENTS

This section presents data review requirements for base, neutral and acid extractable semivolatile organic analysis conducted using the CLP SOW (EPA 1988a). The method of analysis is based on a modification of Method 625 from 40 CFR Part 136 to analyze organic compounds listed on the CLP target compound list (TCL). The data review requirements described herein may be applied to the review of data produced using Method 8240 or Method 8260 (EPA 1986).

4.1 DATA PACKAGE COMPLETENESS

After receipt of the data package and completion of the records management activities detailed in section 2, the reviewer shall organize the data package according to the order of deliverables specified in section 1 of the data validation checklist (Appendix A, Form A-2). Observation of missing data review items that the reviewer deems necessary for completion of the validation shall prompt the reviewer to contact the laboratory for submittal of the needed item. All contacts with the laboratory must be documented on the appropriate form (Appendix B, Form B-8).

4.2 HOLDING TIMES

Review the chain-of-custody forms, laboratory reports, quantitation sheets, extraction records and chain-of-custody for date of sample collection and analysis. All samples must be collected in the required container, protected from light and shipped and stored at 4°C until the time of analysis. Sample extraction must be completed within 7 days of sample collection and analysis completed within 40 days of extraction. Calculate the holding times as follows: extraction holding time = sample extraction date - sample collection date; analysis holding time = sample analysis date - sample extraction date. Complete the holding time summary form (Appendix B) noting the sample collection, extraction and analysis dates. If the criteria were not met, qualify all associated data as estimated (J for detects, UJ for non-detects).

4.3 INSTRUMENT TUNING AND CALIBRATION

Review the laboratory data to verify the laboratory has tuned and calibrated the instrument prior to sample analysis and periodically through the sample analysis batch. Criteria for the review of tuning and calibration data are provided in sections 4.3.1 through 4.3.3.

4.3.1 GC/MS Tuning

Review the tuning reports, mass listings and spectra and verify the laboratory has conducted an acceptable tune using decafluorotriphenylphosphine (DFTPP) for every 12h time period in which samples were analyzed. Using the tuning criteria listed in Table 4-1, check for calculation and transcription errors and the proper reporting of significant figures. Check for errors in calculation of percent abundance values using the mass abundances values reported in the raw data. For example, calculate the percent abundance for m/z 51 relative to m/z 198 using the following formula: % abundance = (relative abundance of m/z 51 / relative abundance of m/z 198) x 100. If the ion abundance criteria are not met, check that the ion ratios for m/z 198/199 and 442/443 and the relative abundances of m/z 68, 70, 197 and 447 are within specification. In addition, check that the expanded criteria for m/z 51, 127, 275, 365 and 442 are within the specifications shown in Table 4-1. If the tuning criteria are not met but the tuning is within the expanded criteria qualify associated data as estimated (J for detects, UJ for non-detects). If all tuning criteria are missed, qualify associated data as unusable (R).

4.3.2 Initial Calibration

Review the calibration forms, quantitation reports and chromatograms to verify the laboratory has conducted an acceptable five-point calibration in accordance with the CLP SOW prior to sample analysis. Verify that all RRF values are ≥0.05, all RSD values are ≤30% and check for calculation errors. Recalculate at least 20% of the individual and average RRF values and RSD values using the following formulas:

- RRF = $(A_x \times C_{is}) / (A_{is} \times C_x)$ where,

  A_x = the area of the characteristic ion for the compound.
  A_{is} = the area of the characteristic ion for the specified internal standard (see EPA 1988a).
  C_x = the concentration of the compound (ng/ul).
  C_{is} = the concentration of the specified internal standard (see EPA 1988a).

- Average RRF = (Sum of individual RRF values) / number of individual RRF values.

- RSD = (Standard Deviation of the individual RRF values) / (Mean of the individual RRF values).

Table 4-1. Semivolatile Organic GC/MS Tuning Criteria

MASS	ION ABUNDANCE CRITERIA (CLP SOW REQUIREMENTS)	EXPANDED CRITERIA (VALIDATION REQUIREMENTS)
51	30.0 - 60.0% of mass 198	22.0 - 75.0% of mass 198
68	Less than 2.0% of mass 69	
69	Mass 69 relative abundance	
70	Less than 2.0% of mass 69	
127	40.0 - 60.0% of mass 198	30.0 - 75.0% of mass 198
197	Less than 1.0% of mass 198	
198	Base Peak, 100% relative abundance	
199	5.0 - 9.0% of mass 198	
275	10.0 - 30.0% of mass 198	7.0 - 37.0% of mass 198
365	Greater than 1.00% of mass 198	Greater than 0.75% of mass 198
441	Present, but less than mass 443	
442	Greater than 40.0% of mass 198	Greater than 30.0% of mass 198
443	17.0 - 23.0% of mass 442	

(Source: EPA 1988a)

Complete the calibration data summary form (Appendix B) summarizing the compounds that exceed the criteria and the affected samples. If any RRF value is out of specification qualify all associated detected results as estimated (J) and all non-detects as unusable (R). If any RSD value is out of specification qualify all associated data as estimated (J for detects, UJ for non-detects).

4.3.3 Continuing Calibration

Review the continuing calibration forms, quantitation reports and chromatograms to verify the laboratory has conducted an acceptable calibration check in accordance with the CLP SOW for every 12h analytical period in which samples were analyzed. Verify that RRF values are ≥0.05, %D values are ≤25%, and check for calculation errors using the formula specified in section 3.3.3.

Complete the calibration data summary form (Appendix B) summarizing the compounds that exceed the criteria and the affected samples. If any RRF value is out of specification qualify all associated detected results as estimated (J) and all non-detects as unusable (R) and if any %D is out of specification qualify all associated data as estimated (J for detects, UJ for non-detects).

4.4 BLANKS

Blanks are analyzed as a means of determining contamination introduced by the laboratory or sampling operations. Contamination may be introduced from the sample handling, sample processing, sample containers, sampling procedures and sample equipment. Prior to beginning the review, complete the blank and sample data summary form (Appendix B) summarizing the detected compounds in all samples and blanks.

4.4.1 Laboratory Blanks

Review the report forms, quantitation reports and chromatograms and verify the laboratory has conducted an acceptable method blank analysis per matrix and extraction batch. The method blank analysis must be conducted after the calibration standard analyses and may contain less or equal to five times the CRQL for common laboratory contaminants such as phthalate esters.

Positive sample results for common laboratory contaminants less than 10 times the blank concentration (less than 5 times for other contaminants) are qualified by elevating the quantitation limit according to the following examples:

- If the sample result is greater than the Contract Required Quantitation Limit (CRQL), but is less than five or ten times the associated highest blank result, the result is qualified as non-detected (U) at the reported concentration.

- If the sample result is less than the CRQL and is less than five or ten times the associated highest blank result, the result value is raised to the CRQL level and qualified as non-detected (U).

- If the sample result is greater than the CRQL and greater than five or ten times the associated highest blank result, no qualification is necessary.

Unidentified TIC compounds in the samples and blanks with retention times that are within one minute of each other and less than five times the highest concentration in any blank contaminant must be considered non-detects (U).

4.4.2 Field Blanks

Following the review of laboratory blank data, review the chain of custody and sample analysis request documentation to identify the field blanks prior to beginning the review. If necessary contact the WHC project coordinator to obtain the necessary information.

4.4.1.1 Equipment Blanks

Review the report forms, quantitation reports and chromatograms. If positive results are reported, it may indicate that decontamination procedures were inadequate or that contamination was inherent to the equipment used and the WHC project coordinator should be notified. Qualify any associated data as non-detected (U) for all positive results that are less than five times the highest valid field blank result.

4.4.1.2 Trip Blanks

Review the field sampling documentation, as necessary to identify any trip blanks. Review the report forms, quantitation reports and chromatograms. Qualify any associated data as non-detected (U) for all positive results that are less than five times the highest valid trip blank result.

4.5 ACCURACY

Laboratory performance and compliance with project-specific and analytical accuracy requirements is determined by a review of surrogate and matrix spike results. The laboratory should conduct at least one matrix spike and matrix spike duplicate analysis per

matrix for each SDG and all samples and blanks shall be spiked with surrogate compounds. A list of the surrogate and matrix spiking compounds and concentrations are found in the CLP SOW, (EPA 1988a).

4.5.1 Surrogate Recovery

Review the surrogate summary forms, quantitation reports and chromatograms, check all surrogate results for calculation errors and verify surrogate recoveries are within the specifications outlined in the CLP SOW (EPA 1988a). Recalculate surrogate recoveries using the following formula: $\%R = (Q_d/Q_a) \times 100$ where, %R = percent recovery, Q_d = quantity of surrogate determined from the analysis, and Q_a = the quantity of surrogate added to the sample. If calculation errors are noted contact the laboratory for clarification and submittal of correct data if necessary. Complete the accuracy data summary form (Appendix B) for all surrogates that exceed the criteria, note the affected samples and qualify associated sample results as estimated (J or UJ) for any two surrogates out of specification but greater than 10%. Qualify all associated detected results as estimated (J) and non-detects as unusable (R) for any surrogate recoveries below 10%. If method blank surrogates are out of specification and associated sample surrogates are acceptable, no qualification is necessary; however, the laboratory should be contacted for an explanation. If method blank and associated sample surrogates are out of specification, contact the laboratory for clarification and document results of such discussions in the validation report.

4.5.2 Matrix Spike Recovery

Review the matrix spike summary forms, quantitation reports and chromatograms, check for calculation errors and verify the laboratory has conducted at least one MS/MSD analysis for each sample delivery group using the requirements specified in the CLP SOW (EPA, 1988a). Recalculate all MS/MSD recoveries using the following formula:

- MS percent recovery = [(SSR - SR) / SA] x 100 where,

  SSR = spiked sample result,
  SR = sample result, and
  SA = spike concentration added from the spiking mixture.

Complete the accuracy data summary form (Appendix B) for MS/MSD compounds that are out of specification. Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the results in the validation narrative. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

4.5.3 Performance Audit Samples

Contact the WHC project coordinator to obtain information regarding the identity and composition of any performance audit sample(s) submitted with the sample batch. Review the report forms, quantitation reports and chromatograms, calculate the percent recovery of each spiked compound and compare to the quality control limits specified by the supplier of the sample. Complete the accuracy data summary form (Appendix B) noting the compounds that exceed the published limits. If the sample results are outside the control limits, contact the laboratory for an explanation and document the details in the validation narrative.

4.6 PRECISION

The precision of the overall measurement system is determined by the reproducibility of MS/MSD analyses, field duplicates and split samples.

4.6.1 Matrix Spike/Matrix Spike Duplicate Samples

Review the MS/MSD reports, quantitation reports and chromatograms, verify the laboratory has conducted at least one MS/MSD analysis for the sample delivery group and check for calculation errors. Recalculate all MS/MSD RPD values using the following formula:

- MS/MSD RPD = $|(D1 - D2)| / [(D1 + D2) / 2] \times 100$ where,

  RPD = relative percent difference,
  D1 = MS value, and
  D2 = MSD value (duplicate).

Complete the precision data summary form (Appendix B) documenting those MS/MSD compounds that exceed the criteria, and note the affected samples. Review the MS/MSD results in conjunction with other QC data such as field duplicates and note the results in the validation narrative. If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

4.6.2 Field Duplicate Samples

Review the chain of custody and sample analysis request documentation to identify field duplicates and their corresponding samples, report forms, quantitation reports and chromatograms. Calculate the RPD of the positive results for each compound using the

CRQL if one of the results is a non-detect. Since precision criteria have not been developed by EPA, the inorganic analysis duplicate criteria have been applied to the evaluation of organic analysis field duplicate results. RPD limits for field duplicates where both results are greater than five times the CRQL are 20% for waters and 35% for soils and when one or both results are less than five times the CRQL the limits are ±CRQL for waters and ±2xCRQL for soils. Complete the precision data summary form (Appendix B) documenting the results and RPD values for all detected compounds and note the results of the field duplicate analyses in the validation narrative.

4.6.3 Field Split Samples

Review the chain of custody and sample analysis request documentation to identify field split and their corresponding samples, report forms, quantitation reports and chromatograms. Contact the WHC project coordinator to obtain information about the sample results from participating referee laboratories. Refer to Section 4.6.2 for review and reporting requirements.

4.7 SYSTEM PERFORMANCE

Review report forms, chromatograms and quantitation reports for evidence of RIC baseline anomalies, retention time shifts, extraneous peaks, low resolution and peak anomalies. Check that positive results are not affected by abrupt changes in baseline caused by leaks in the MS system or GC column bleed. In addition look for positive results affected by coeluting compounds and ensure that the detected compound is resolved by at least ≥25%. If it is in the reviewers informed professional judgement that quantitative sample results may be biased due to system performance anomalies this judgement must be noted in the validation narrative and the affected results qualified accordingly.

4.7.1 Internal Standards Performance

Review the report forms, quantitation reports and chromatograms, verify that area counts and retention times comply with CLP SOW (EPA 1988a) requirements and check for calculation and transcription errors. If area counts are outside the limits of +100 to -50 percernt qualify associated sample results as estimated (J for detects, UJ for non-detects). If area counts are outside the limits and relative retention time criteria are > ± 30 seconds qualify all non-detects as unusable (R).

4.8 COMPOUND IDENTIFICATION AND QUANTITATION

Review the quantitation reports, chromatograms, and spectra for laboratory standards, samples and blanks to verify positive identification of TCL compounds. Verify that retention times for positive results in samples are within ±0.06 relative retention time units of the associated calibration standard and review chromatograms to verify that all peaks are identified. Compare sample mass spectra to laboratory-generated standard spectra for compliance with the following criteria:

- All ions present in the standard at a relative intensity of 10% or greater are present in the sample spectrum,
- Relative intensities of standard and sample spectra agree within 20%, and
- Ions greater than 10% in the sample spectrum and not in the standard are identified and explained.

Qualify all affected positive results as follows:

- If the retention time criteria and mass spectral criteria are exceeded, qualify the results as unusable (R) and noted in the validation narrative.
- If the reviewer determines that incorrect identifications were made as a result of cross-contamination between analyses then affected data should be qualified as unusable (R) and noted in the validation narrative.

4.8.1 Reported Results and Quantitation Limits

Review the report forms, quantitation reports and chromatograms, verify that correct internal standards, quantitation ions, and RRF values were used for quantitation and check for calculation errors. In addition, review sample dilutions and dry weight factors to verify accurate adjustments for CRQL values. Recalculate at least 20% of all results using the following formula:

- Water samples, $\mu g/L = (A_x \times I_s \times V_t) / (A_{is} \times RRF \times V_o \times V_i)$ where,

  A_x = area of the quantitation ion for the compound,
  A_{is} = area of the quantitation ion for the specified internal standard,
  RRF = relative response factor for the compound from the daily standard,
  I_s = amount of the specified internal standard in nanograms (ng),
  V_t = Volume of total extract in microliters, taking into account any dilutions,
  V_o = the volume of water extracted in milliliters, and
  V_i = the volume of extract injected in microliters.

- Soil samples, $\mu g/Kg = (A_x \times I_s \times V_t) / (A_{is} \times RRF \times V_i \times W_s \times D)$

  A_x, I_s, A_{is}, RRF, V_i and V_t = same as for waters above
  W_s = the weight of sample extracted in grams
  D = the percent solids

Determine if the laboratory was able to meet the project specific CRQL goals. In the absence of known or suspected analytical interferences, if the laboratory was unable to determine any compound within five times the CRQL, the laboratory must be contacted for clarification; results of such discussions must be noted in the validation narrative.

4.8.2 Tentatively Identified Compounds (TIC)

Chromatographic peaks may be present in an analysis that are not TCL analytes, surrogates, or internal standards and are considered tentatively identified compounds (TIC) and must be qualitatively identified by the laboratory.

Verify that spectral library searches were conducted for at least 20 or less candidate TIC in accordance with the CLP SOW (EPA 1988a). Qualify as non-detects all compounds including common laboratory contaminants if present in the blanks using the blank review criteria in Section 4.4. A list of common laboratory contaminants often detected and reported as TIC contaminants are presented in Table 3-2.

In addition, the assessment of blanks should include an examination for TIC peaks present but not reported in the blanks due to the peaks being less than 10% of the associated internal standard height. All TIC peaks should be evaluated against the following criteria and considerations:

- Major ions (> 10% relative intensity) in the reference spectra should be present in the sample spectra;
- Relative intensities of major ions in the sample spectra should be within ±20% of the associated reference spectra;
- Molecular ions in the reference spectra should be present in the sample spectra;
- Ions present in the sample but not in the reference spectra should be reviewed for the possibility of interferences caused by co-elution of other TIC or possibly TCL compounds;
- If the TIC is not found in the blanks but is a suspected laboratory contaminant the result should be qualified as unusable (R);

- If the library search reveals more than one acceptable compound match the result may be qualified as a non-specific isomer; and

- If the sample(s) contain groups of TIC results that are similar isomers the reviewer should summarize and report all similar isomers as total, such as all alkanes reported as total alkanes or all unknowns reported as total unknowns.

If the reviewer determines that TIC identification is in error the associated sample results should be qualified as non-detects (U) or unusable (R). If the TIC identification is determined to be valid, the results are to be qualified as presumptive and estimated (JN).

4.9 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A) and qualify affected data as determined from the review on the data qualification summary (Appendix B). Briefly summarize any technical problems associated with the data, and the overall quality and useability of the data in the validation narrative according to the requirements of Section 10.

5.0 PESTICIDE AND PCB DATA REVIEW REQUIREMENTS

This section presents data review requirements for extractable pesticide and polychlorinated biphenyl compounds conducted using the CLP SOW (EPA 1988a). The method of analysis is based on a modification of Method 608 from 40 CFR Part 136 to analyze organic compounds listed on the CLP target compound list (TCL). The data review criteria described herein may be applied to data produced using Method 8080 or Method 8081 (EPA 1986).

5.1 COMPLETENESS AND CONTRACT COMPLIANCE

After receipt of the data package and completion of records management activities detailed in section 2, the reviewer shall organize the data package according to the order of deliverables specified in section 1 of the data validation checklist (Appendix A, Form A-3). Missing data review items that the reviewer deems necessary for completion of the validation shall require the reviewer to contact the laboratory for submittal of the needed item. All contacts with the laboratory must be documented on the appropriate form (Appendix B, Form B-8).

5.2 HOLDING TIMES

Review the chain-of-custody forms, the laboratory reports and the extraction worksheets for the date and time of sample collection, extraction, and analysis. Samples must be collected in the proper containers, protected from light and maintained at 4°C until the time of analysis. Samples must be extracted within 7 days from time of collection and analyzed within 40 days of extraction date. Calculate the holding times as follows: extraction holding time = sample extraction date - sample collection date; analysis holding time = sample analysis date - sample extraction date. The holding time summary form (Appendix B) must be completed. If any holding time is missed, qualify all associated data as estimated (J for detects, UJ for non-detects).

5.3 INSTRUMENT PERFORMANCE AND CALIBRATIONS

The gas chromatograph must pass specific criteria prior to the analysis of samples to ensure maximum instrument sensitivity and chromatographic resolution specific to pesticide and polychlorinated biphenyl (PCB) compounds. Review the appropriate instrument performance data and calibration data which includes the raw data sheets, analyst notebook records and quantitation reports to ensure that the laboratory has properly calibrated the gas chromatograph prior to analysis of the samples.

5.3.1 Instrument Performance

Review the evaluation standards, calibration standards and quantitation reports to verify that the following instrument performance criteria were met.

- Review DDT retention times and check that all are greater than 12 minutes on packed columns and check that resolution between peaks is ≥25%. Calculate resolution for at least 20% of the reported results using the following formula:

  Resolution = $(P_v / P_h) \times 100$ where,

  P_v = the peak height of the valley of the larger peak.
  P_h = the peak height of the smaller peak being resolved.

  If the DDT retention time criteria are not met and resolution is not adequate, qualify associated data as unusable (R).

- Review the calibration standard summary for correctly reported retention time windows and check that all standards fall within the specified ranges. Calculate retention time windows using the procedures specified in the CLP SOW (EPA, 1988a)

  If the standards do not fall within the retention time windows associated sample results after the last in-control may be affected. If no peaks are present within the retention time window of the deficient pesticide of interest no qualification is necessary. If peaks are present in samples within the retention time window a review is made of the raw data to determine expanded retention time windows for the pesticide of interest from available standards analyzed within the 72h period in which the affected samples were analyzed. If all standards and matrix spikes fall within the expanded windows then no qualification of sample results is necessary. If all standards and matrix spikes do not fall within the expanded windows then all affected sample results are qualified as unusable (R).

- Review the DDT and endrin breakdowns in all applicable standards and check calculations on 20% of the reported results using the following formula:

  Percent breakdown = (Total degradation peak area) / (Total peak area) x 100 where, the total degradation peak area is the sum of the peak areas for DDE and DDD or endrin aldehyde and endrin ketone and the total peak area is the sum of all associated peak areas for DDE, DDD and DDT or endrin, endrin aldehyde and endrin ketone.

If the DDT percent breakdown exceeds 20%, qualify all detected results for DDT as estimated (J) and all non-detects as unusable (R) if DDD and DDE are detected. In addition qualify all detected results for DDD or DDE as presumptive and estimated (NJ).

If the endrin breakdown exceeds 20%, qualify all detected results for endrin as estimated (J) and all non-detects as unusable (R) if endrin aldehyde or endrin ketone are detected. In addition qualify all detected results for endrin ketone as presumptive and estimated (NJ).

- Review and verify the percent difference in retention time for dibutylchlorendate (DBC) in all standards and samples is ≤2.0% for packed columns, ≤0.3% for capillary columns and ≤1.5% for wide-bore capillary column analysis. Calculate the percent difference values in all samples and standards with the following formula:

  $\%D = RT_i - RT_s / RT_i \times 100$ where,

  RT_i = the absolute retention time of DBC in the initial standard (Evaluation mixture A).

  RT_s = the absolute retention time of DBC in the sample and subsequent standards.

  If the retention time criteria are exceeded for DBC and the shift is occurring repeatedly in samples and standards, qualify the sample analysis as unusable (R).

5.3.2 Calibrations

Review the evaluation standards, chromatograms, run logs and quantitation reports and verify initial calibration linearity, analytical sequence and continuing calibration percent differences as detailed below.

- Verify linearity by calculating 20% of the initial calibration factors, mean calibration factors and %RSD values for aldrin, endrin, DDT and DBC using the equations provided in the CLP SOW (EPA 1988a). Verify that the %RSD values are less than 10% for quantitation columns only and if the column is used only for surrogate quantitation the DBC is only required to meet the 10% criterion. In addition if the DDT series or toxaphene were identified verify that a three point calibration was conducted.

  If the linearity criteria are exceeded, qualify associated detected results as estimated (J).

- Verify that all standards were analyzed at the beginning of each 72 hour period. For confirmation analyses verify that evaluation mixes A, B and C were analyzed for the calibration curve, that the confirmation standards were repeated after every 5 samples and that evaluation mix B was repeated after every 10 samples.

  If the proper confirmation standards were not analyzed, and continuing calibration criteria were not met for either quantitation or confirmation standards qualify associated detected results as unusable (R).

- Verify that the percent difference between each subsequent standard and the standard at the beginning of the analytical sequence is less than 15% for the quantitation analyses. Recalculate 20% of the %D values from the raw data and compare to the reported results.

  If the %D criteria are exceeded qualify associated detected results (for quantitation analyses only) as estimated (J).

Finally, complete the calibration data summary form (Appendix B) for all values that exceed the criteria noting the samples that require qualification.

5.4 BLANKS

The blank data results are reviewed to assess the extent of contamination introduced through sampling, extraction and analysis. Prior to completing review of the blanks, complete the blank and sample data summary form (Appendix B) by summarizing detected results in all samples and blanks.

5.4.1 Laboratory Blanks

Review the report forms, quantitation reports and chromatograms and verify the laboratory has conducted an acceptable method blank analysis for each instrument, matrix, concentration level and extraction batch. The method blank analysis must be conducted in accordance with the analytical sequence outlined in the CLP SOW (1988a) and may contain target compounds less than or equal to the CRQL. If the laboratory has failed to analyze a method blank for each matrix, instrument and extraction batch, contact the laboratory for submittal of the proper data.

Qualify all associated positive sample results as non-detects (U) that are less than five times the highest amount in any blank.

5.4.2 Field Blanks

Following the review of laboratory blanks, review the chain of custody and sample analysis request documentation to identify the field blanks prior to beginning the review and contact the WHC project coordinator to obtain the necessary information. Review the report forms, quantitation reports and chromatograms. If positive results are reported in the equipment blanks, it may indicate that decontamination procedures were inadequate in the sampling process, or that contamination was inherent to the equipment used and the project coordinator should be notified. Qualify any associated data as non-detected (U) for all positive results that are less than five times the highest valid field blank result.

5.5 ACCURACY

Laboratory performance and compliance with project specific accuracy requirements is determined by a review of surrogate recovery, matrix spike/matrix spike duplicate recovery and if applicable, performance audit sample results. The laboratory should conduct matrix spike and matrix spike duplicate analysis for every matrix and extraction batch of 20 samples or less. Surrogates must be added to all samples at concentrations specified in the CLP SOW (EPA 1988a).

5.5.1 Surrogate Recovery

Review the surrogate summary forms, quantitation reports and chromatograms, check for calculation errors and verify the DBC surrogate recoveries are within the limits of 24% to 154% for waters, and 20% to 150% for soils. Complete the accuracy data summary form (Appendix B) for all surrogates that exceed the criteria and note the affected samples. Qualify all associated sample results as estimated (J or UJ) for surrogates out of specification. If the surrogate was not detected (0% recovery) in the sample qualify associated data as unusable (R). If method blank surrogates are out of specification and associated sample surrogates are acceptable, no qualification is necessary; however, the laboratory should be contacted for an explanation. If method blank and associated sample surrogates are out of specification, contact the laboratory for clarification and document subsequent discussions in the validation report.

5.5.2 Matrix Spike Recovery

Review the matrix spike summary forms, quantitation reports and chromatograms, check for calculation errors and verify the laboratory has conducted at least one MS/MSD analysis per matrix for each sample group using the requirements specified in the CLP SOW (EPA, 1988a). Complete the accuracy data summary form (Appendix B) for MS/MSD compounds that exceed the criteria and note the affected samples. Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the

results in the validation narrative. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

5.5.3 Performance Audit Samples

Contact the WHC project coordinator to obtain information regarding the identity and composition of any performance audit sample(s) submitted with the sample batch. Review the report forms, quantitation reports and chromatograms, calculate the percent recovery of each spiked compound and compare to the quality control limits specified by the supplier of the sample. Complete the accuracy data summary form (Appendix B) noting the compounds that exceed the published limits. If the sample results are outside the control limits, contact the laboratory for clarification and document subsequent discussions in the validation narrative.

5.6 PRECISION

The review of field and laboratory precision provides information necessary to evaluate the reproducibility of laboratory results and to determine whether sampling activities are adequate for the collection of consistent data.

5.6.1 Matrix Spike/Matrix Spike Duplicate Samples

Review the MS/MSD reports, quantitation reports and chromatograms, verify the laboratory has conducted at least one MS/MSD analysis for the sample delivery group and check for calculation errors. Recalculate all MS/MSD RPD values using the following formula:

- MS/MSD RPD = $|(D1 - D2)| / [(D1 + D2) / 2] \times 100$ where,

  RPD = relative percent difference,
  D1 = MS value, and
  D2 = MSD value (duplicate).

Complete the precision data summary form (Appendix B) documenting those compounds that exceed the criteria and note the affected samples. Review the MS/MSD results in conjunction with other QC data such as field duplicates and not the results in the validation narrative. If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

5.6.2 Field Duplicate Samples

Review the field sample documentation to identify field duplicates and their corresponding samples, report forms, quantitation reports and chromatograms. Calculate the RPD of the positive results for each compound using the CRQL if one of the results is a non-detect. RPD limits for field duplicates where both results are greater than five times the CRQL are 20% for waters and 35% for soils. When one or both results are less than five times the CRQL, the RPD limits are ± CRQL for waters and ±2x the CRQL for soils. Complete the precision data summary form (Appendix B) documenting the results and RPD values for all detected compounds and note the results of the field duplicate analyses in the validation narrative.

5.6.3 Field Split Samples

Contact the WHC project coordinator for the identity and results of field split samples. Calculate the field split RPD values for detected compounds, summarize on the precision data summary form (Appendix B) and follow the review and reporting requirements as specified in section 5.6.2.

5.7 COMPOUND IDENTIFICATION AND QUANTITATION

Qualitative criteria have been established to minimize false positives and negatives in the reporting of pesticide/PCB data. These criteria include compliance with retention time window criteria on dissimilar gas chromatography (GC) columns and gas chromatography/mass spectrometry (GC/MS) confirmation if the sample concentration for any single pesticide or PCB compound exceeds 10 ppm in the sample extract.

5.7.1 Compound Identification

After review of the quantitation reports, standards data and dual column identification data, the reviewer must determine if the following criteria have been met for TCL compound identification.

- Review the reported results and raw data and verify that positive results are within the retention time windows. If the qualitative criteria are not met qualify detects as non-detects as follows: If the misidentified peak is outside the retention time windows and no interferences are noted report the CRQL and if the misidentified peak interferes with a target peak then the report value is qualified as estimated and non-detected (UJ);

- Review the raw data to determine that positive results were analyzed on dissimilar columns and if not, reject affected data (R);

- Verify from the raw data that a 3% OV-1 column was not used to confirm both dieldrin and DDE if detected. If the 3% OV-1 column was used for confirmation of dieldrin and DDE reject associated data (R);

- Verify from the raw data that multipeak pesticides (chlordane and toxaphene) and PCBs match the standard chromatograms. If quantitation and confirmation are questionable all affected data should be qualified as presumptive and estimated (NJ); and

- If GC/MS confirmation was required but not conducted, contact the laboratory for explanation and note in the validation narrative.

5.7.2 Reported Quantitation Limits

After reviewing quantitation reports, sample preparation logs, extraction worksheets and case narratives recalculate results to ensure CRQL values were adjusted for sample dilution, sample concentrations, splits, clean-up activities and dry weight factors. Determine if the laboratory was able to meet the project specific CRQL goals. In the absence of known or suspected analytical interferences, if the laboratory was unable to determine any compound within five times the CRQL, the laboratory must be contacted for clarification; subsequent discussion should be documented in the validation narrative.

5.8 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A) in accordance with the requirements of Section 10. Briefly summarize any technical problems associated with the data, and prepare a narrative report that summarizes data acceptability in terms of the project data quality objectives. Qualify affected data as determined from the review on the data qualification summary (Appendix B).

6.0 HERBICIDE DATA REVIEW REQUIREMENTS

This section presents specific review requirements for chlorinated herbicide analyses for the compounds 2,4-D and 2,4,5-TP (Silvex). The analytical requirements specified for these analyses are contained in EPA Method 8150 (EPA 1986) or 509B (APHA 1985). This analysis requires solvent extraction of a sample aliquot followed by hydrolysis and esterification of the herbicide compounds, concentration of the solvent extract and injection into a GC with ECD detection. Specific data review requirements for herbicide analyses have not been developed by EPA; hence, in the absence of such requirements the CLP SOW (EPA 1988a) data review requirements specified for pesticide and PCB compounds and the method performance requirements are applied here.

6.1 GENERAL REQUIREMENTS

Completion of herbicide data review will require the reviewer to have the following reference available in addition to the materials specified in Section 2.

- Method 8150, Chlorinated Herbicides, SW-846, Revision 0, September 1986.

6.2 DATA PACKAGE COMPLETENESS

After receipt of the data package and completion of records management activities detailed in section 2, the reviewer shall organize the data package according to the order of deliverables specified in section 1 of the data validation checklist (Appendix A, Form A-4). Observation of missing data review items that the reviewer deems necessary for completion of the validation shall prompt the reviewer to contact the laboratory for submittal of the needed item. All contacts with the laboratory must be documented on the appropriate form (Appendix B, Form B-8).

6.3 HOLDING TIMES

Review the chain-of-custody forms, the laboratory result reports and extraction data sheets. All samples must be collected in the proper containers, protected from light, shipped, received and stored at 4°C until the time of analysis. Samples must be extracted within 7 days of collection and analyzed within 40 days of extraction. Complete the holding time summary form (Appendix B) noting the samples, extraction and analysis dates. If the criteria were not met, qualify all associated data as estimated (J for detects, UJ for non-detects).

6.4 INSTRUMENT CALIBRATION

Review the laboratory instrument calibration data to ensure the laboratory has acceptably calibrated the GC prior to sample analysis and periodically during analysis of samples.

6.4.1 Initial Calibration

Review the calibration forms, quantitation reports and chromatograms to verify the laboratory has conducted an acceptable initial calibration in accordance with method requirements prior to sample analysis. Verify calculations according to the method requirements and check that the RSD of the calibration factors for 2,4-D and 2,4,5-TP (Silvex) are less than 20%. Complete the calibration data summary form (Appendix B) for compounds that exceed the criteria. If the RSD criteria are not met, qualify all associated data between the initial calibration and the nearest continuing calibration as estimated (J for detects, UJ for non-detects).

In addition, determine the retention time windows for all calibration standards for comparison to continuing calibrations and sample results. Since retention time criteria have not been established for herbicides the procedures specified in the CLP SOW for pesticide/PCBs shall be used (EPA, 1988a).

6.4.2 Continuing Calibrations

Review the continuing calibration reports, quantitation reports and chromatograms to verify the laboratory has conducted an acceptable daily calibration check. Verify the calculations and check that response factor (RF) values are within 15% of the initial calibration values. Check that the continuing calibration compounds elute within the retention time windows determined in Section 6.4.1 and complete the calibration data summary form (Appendix B) summarizing the compounds that exceed the criteria and the affected samples. If the percent difference criteria or retention time windows are not met, qualify all associated data as estimated (J for detects, UJ for non-detects).

6.5 BLANKS

Review the laboratory result reports, chromatograms, quantitation reports and analyst notebook sheets. Blanks are analyzed as a means of determining contamination introduced by the laboratory or sampling operations and may be introduced from the sample handling and processing, sample containers, field sampling procedures and equipment. Prior to beginning the review, complete the blank and sample data summary form (Appendix B) summarizing the detected compounds in all samples and blanks.

6.5.1 Laboratory Blanks

Review the results for the laboratory blanks, recalculate and check that results are reported properly. Verify the laboratory has conducted at least one method blank analysis per sample delivery group and per matrix. The method blank(s) may contain less than the CRQL of any target compound or interferant. Qualify samples that contain less than five times the highest laboratory blank concentration as non-detects (U). If the laboratory blanks contain herbicides and the samples do not, the laboratory should be contacted for an explanation as this may indicate a laboratory contamination problem. Document all subsequent discussions in the validation narrative.

6.5.2 Field Blanks

Following the review of laboratory blanks, review obtain copies of the field sampling documentation to identify the field blank samples and sample types. Review the results for the field blanks; if positive results are reported, it may indicate that decontamination procedures were inadequate or that contamination was inherent to the equipment used, and the project coordinator should be notified. Qualify any associated data as non-detected (U) for all positive results that are less than five times the highest valid field blank result.

6.6 ACCURACY

Laboratory performance and compliance with project specific and analytical accuracy requirements is determined by a review of surrogate and matrix spike results. The laboratory should conduct at least one matrix spike and matrix spike duplicate analysis for each SDG; all samples and blanks should be spiked with an appropriate surrogate.

6.6.1 Surrogate Recovery

Surrogate results aid in the determination of laboratory accuracy on each sample and blank. The laboratory should spike all samples and blanks with an appropriate surrogate. The surrogate most often used for herbicide analysis is the compound 2,4-DB. Review the laboratory results, quantitation reports and chromatograms to determine that the surrogate recoveries for all samples in the SDG are within the pesticide/PCB limits of 24% to 154% (EPA 1988a). Complete the accuracy data summary form (Appendix B) for surrogates that exceed the criteria and qualify all associated data as estimated (J for detects, UJ for non-detects) if surrogate recoveries are out of specification. If surrogate recoveries are zero, qualify all detects as estimated (J) and non-detects as unusable (R), and note in the validation narrative.

6.6.2 Matrix Spike Recovery

Review the laboratory results, chromatograms and quantitation data and verify the absence of calculation and transcription errors. Advisory limits for herbicide matrix spikes are recommended at 40% to 130% based on the average upper and lower limits for water and soil established for pesticides in the CLP SOW (EPA 1988a). Complete the accuracy data summary form for compounds that exceed the criteria. Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the results in the validation narrative. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

6.6.3 Performance Audit Samples

Contact the WHC project coordinator to obtain information regarding the identity and composition of any performance audit sample(s) submitted with the sample batch. Review the report forms, quantitation reports and chromatograms, calculate the percent recovery of each spiked compound and compare to the quality control limits specified by the supplier of the sample. Complete the accuracy data summary form (Appendix B) noting the compounds that exceed the published limits. If the sample results are outside the control limits, contact the laboratory for clarification and document all subsequent discussions in the validation report narrative.

6.7 PRECISION

The review of field and laboratory precision provides information on the laboratory reproducibility and whether sampling activities are adequate to acquire consistent samples.

6.7.1 Matrix Spike/Matrix Spike Duplicate Samples

Check the laboratory results and raw data sheets and verify the absence of calculation and transcription errors. Advisory limits for herbicide matrix spike duplicates are recommended at 50% for waters and soils based on the average RPD values specified for pesticides in the CLP SOW (EPA 1988a). Complete the precision data summary form (Appendix B) documenting those MS/MSD compounds that exceed the criteria and note the affected samples. Review the MS/MSD results in conjunction with other QC data such as field duplicates and not the results in the validation narrative. If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

6.7.2 Field Duplicate Samples

Review the chain of custody and sample analysis request documentation to identify field duplicates and their corresponding samples, report forms, quantitation reports and chromatograms. Calculate the RPD of the positive results for each using the CRQL if one of the results is a non-detect. Since precision criteria have not been developed by EPA, the inorganic analysis duplicate criteria have been applied to the evaluation of herbicide analysis field duplicate results. RPD limits for field duplicates where both results are greater than five times the CRQL are 20% for waters and 35% for soils. When one or both results are less than five times the CRQL the RPD limits are ±CRQL for waters and ±2xCRQL for soils. Complete the precision data summary form (Appendix B) documenting the results and RPD values for all detected compounds; note the results of the field duplicate analyses in the validation narrative.

6.7.3 Field Split Samples

Obtain information related to the identity of field split samples from the WHC project coordinator and follow the review and reporting requirements specified in Section 6.7.2 for field duplicates.

6.8 COMPOUND IDENTIFICATION AND QUANTITATION

Review the calibration and sample data and determine that positive results are unaffected by interferant peaks and are within the retention time windows established in Section 6.4.1. If positive results are not within retention time windows, qualify all detected results as non-detects (U) using the following criteria for assigning the quantitation limit.

- If the misidentified peak is outside the retention time windows and no potential interferences are present, then the compound CRQL is reported.

- If the misidentified peak interferes with the potential detection of a target peak then the reported value is the quantitation limit and the result is qualified as estimated (UJ).

6.8.1 Reported Quantitation Limits

Check that sample results have been calculated properly and that the laboratory has met the project specific CRQL goals for the analysis. Check at least 20% of the reported detected and nondetected values by using the calculation formula provided in EPA Method 8150 or the laboratory standard operating procedures. In the absence of known or suspected analytical interferences, if the laboratory was unable to quantitate within five

times the CRQL, contact the laboratory for clarification and document all subsequent discussions in the validation narrative.

6.9 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A), and qualify affected data as determined from the review on the data qualification summary (Appendix B). Briefly summarize any technical problems associated with the data and prepare a narrative report that summarizes the data acceptability in terms of the project data quality objectives according to the requirements of Section 10.

7.0 DIOXIN/FURAN DATA REVIEW REQUIREMENTS

This section presents specific data review requirements for laboratory analysis of polychlorinated dibenzo-p-dioxin and polychlorinated dibenzofuran compounds. These analyses are normally conducted using the procedures specified in Method 8290 (EPA 1989a).

7.1 GENERAL REQUIREMENTS

The reviewer shall have the following reference available for dioxin/furan data review:

- Method 8290, Determination of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS), Revision 0, October 1989.

7.2 DATA PACKAGE COMPLETENESS

After receipt of the data package and completion of records management activities detailed in section 2, the reviewer shall organize the data package according to the order of deliverables specified in section 1 of the data validation checklist (Appendix A, Form A-5). Missing data review items that the reviewer deems necessary for completion of the validation shall require the reviewer to contact the laboratory for submittal of the needed item. All contacts with the laboratory must be documented on the appropriate form (Appendix B, Form B-8).

7.3 HOLDING TIMES

Review the chain-of-custody forms, laboratory reports, quantitation sheets and extraction data sheets. All samples must be collected in the proper containers and shipped and maintained at 4°C prior to analysis. It is recommended that extractions be completed within 30 days of collection and extracts completely analyzed within 45 days of collection (EPA 1990) however, PCDDs and PCDFs are considered very stable and in some cases holding times may be as high as a year for some matrices. In any case, sample extracts should always be analyzed within 45 days of extraction (EPA 1990). Complete the sample holding time summary form (Appendix B) noting samples that exceed the criteria and qualify all associated data as estimated (J for detects, UJ for non-detects).

7.4 INSTRUMENT PERFORMANCE AND CALIBRATION

The laboratory must demonstrate that system performance and calibration criteria are met before sample analysis may begin. The following sections provide specific data review criteria for instrument calibration and tuning.

7.4.1 GC Column Performance

A GC column performance check should be analyzed at the beginning of each 12h shift in which samples are analyzed. Verify that the following criteria are met:

- Chromatographic separation between 2,3,7,8-TCDD and other unlabeled TCDD isomers are resolved at $\leq$ 25 percent.
- Verify from the raw data that the laboratory has established retention time windows for the PCDD/PCDF isomers in the performance check solution, and
- Verify from the raw data that the laboratory has established proper instrument conditions for the switching of selected ion monitoring (SIM) ions for each isomer series.

If the column performance criteria are not met, reject associated data as unusable (R).

7.4.2 MS Performance

The mass spectrometer instrument must be operated in electron ionization mode and be capable of providing a static resolving power of at least 10,000. Instrument resolving power must be documented by reporting the peak profile of mass 380.9760 for the perfluorokerosene (PFK) tuning compound in accordance with Method 8290 (EPA, 1989a) and a hard copy printout of this MS resolution adjustment must be provided. If the laboratory has failed to achieve the resolution requirements, qualify all associated data as unusable (R).

7.4.3 Initial Calibration

Review the calibration summary data, quantitation sheets and chromatograms and ensure that a five point initial calibration has been successfully completed. Check and recalculate 20% of the RSD values of the mean and standard deviation of the response factors for the labeled and unlabeled standards. The RSD must be ≤20% for the unlabeled standards and ≤30% for the labeled standards. Review the chromatograms to ensure the signal to noise ratio in all calibration runs is ≥2.5 when calculated using the equation provided in Method 8290. Review the calibration summaries and quantitation sheets and ensure the chlorine isotope ratios are within limits. Complete the calibration data summary

form (Appendix B) for compounds that exceed the aforementioned criteria and qualify associated data as estimated (J for detects, UJ for non-detects).

7.4.4 Continuing Calibrations

Review the continuing calibration summary data, quantitation sheets and chromatograms and ensure that a calibration check has been performed during every 12 hour time period in which samples were analyzed and verify the RSD values between the mean initial calibration response factors and continuing calibration response factors are within the limits specified for initial calibrations. Complete the calibration data summary form (Appendix B) for compounds that exceed the criteria and qualify associated data as estimated (J for detects, UJ for non-detects).

7.5 BLANKS

Blanks are analyzed to ensure that sample collection, handling, extraction and analysis procedures are not introducing contamination that may affect the validity of the analytical results. Blanks should be free of dioxin and furan compounds (except octa-substituted congeners). Prior to beginning review of the blank data, complete the blank and sample data summary (Appendix B) summarizing all detected compounds in the samples and blanks.

7.5.1 Laboratory Blanks

At least one laboratory method blank should be analyzed for each matrix with each sample group. Method blanks should not contain greater than 10 percent of the sample quantitation limit for any of the 2,3,7,8-substituted congeners (except the octa-congeners). Review the sample result reports, quantitation sheets and sample extraction data sheets. Qualify positive sample results that are less than five times the highest method blank concentration as non-detects (U).

7.5.2 Field Blanks

Field blanks should be submitted with each sample batch and should include at least one equipment blank to measure the sampling equipment decontamination effectiveness and one trip blank to determine the cleanliness of the sample containers. Obtain information about the identity of field blanks from the project coordinator to include in the data review report. Review the sample result reports, quantitation sheets and sample extraction data sheets. Sample results for specific congeners that are less than five times the highest concentration in the valid field blanks are qualified as non-detects (U).

7.6 ACCURACY

At least one matrix spike and matrix spike duplicate should be analyzed for each matrix for each sample batch. In addition, the identity and composition of any performance audit samples submitted must be obtained from the WHC project coordinator.

7.6.1 Matrix Spike Recovery

Review the matrix spike result reports, quantitation sheets and extraction data sheets and ensure that no transcription or calculation errors are noted. Recalculate all the MS/MSD recoveries using the formula provided in Section 3.5.2. Matrix spike recovery limits have not been established for dioxin and furan analyses so the internal standard recovery requirements of 40 to 120% are recommended (EPA, 1989a). Summarize the compounds exceeding the recovery limits and the associated samples on the accuracy data summary form (Appendix B). Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the results in the validation narrative. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

7.6.2 Performance Audit Samples

Contact the WHC project coordinator to obtain the identity, source and performance criteria for the dioxin performance audit sample. Review the laboratory result reports, quantitation sheets and extraction data sheets. Summarize the results on the accuracy data summary form and note compounds that exceed the published limits. If the sample results fall outside the acceptance limits the laboratory should be contacted for clarification and all subsequent discussions should be documented in the validation narrative.

7.7 PRECISION

Precision of dioxin/furan analyses is assessed by the analysis of matrix spike duplicates, field duplicates and split samples. The following sections provide guidance on the review of these types of analyses.

7.7.1 Matrix Spike/Matrix Spike Duplicate Samples

At least one set of matrix spike/matrix spike duplicate analyses must be conducted for each sample matrix and sample batch. Review the laboratory matrix spike results, quantitation sheets and extraction sheets and check for calculation errors. Relative percent difference limits for MS/MSD samples are 20% (EPA, 1989a). Summarize the compounds

that exceed the 20% criteria and the affected samples on the precision data summary form (Appendix B). Review the MS/MSD results in conjunction with other QC data such as field duplicates and not the results in the validation narrative. If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

7.7.2 Field Duplicate Samples

Contact the WHC project coordinator and obtain the identity and source of the field duplicate samples submitted with the sample batch. At least one set of blind field duplicate samples should be submitted with each batch. Calculate the RPD of the positive results for each compound using the CRQL if one of the results is a non-detect. Since precision criteria have not been developed by EPA, the inorganic analysis duplicate criteria have been applied to the evaluation of dioxin/furan field duplicate results. Criteria for duplicate sample results below five times the CRQL limits are ±CRQL for water samples and ±2x the CRQL for soil samples. RPD limits for results greater than five times the CRQL are 20% for water and 35% for soils. Summarize the field duplicate results on the precision data summary form (Appendix B) and discuss those compounds that exceed the criteria in the validation narrative.

7.7.3 Field Split Samples

Contact the WHC project coordinator and obtain the identity and results of any split samples submitted as part of the sample batch. Review criteria for field splits are the same as described in Section 7.7.2.

7.8 SYSTEM PERFORMANCE

Assess system performance by review of all chromatograms for anomalies such as extraneous peaks, low signal to noise ratios, baseline shifts, peak tailing, low resolution (≤25%) of standards and general instrument stability. Note any anomalies in the summary section of the data validation checklist and contact the laboratory for clarification.

7.8.1 Internal Standards Performance

Review the laboratory reports and quantitation sheets to verify that internal standards recoveries have been calculated and transcribed properly. Percent recovery limits are 40 to 120% (EPA, 1989a). Record compounds that exceed the recovery criteria on the accuracy data summary form (Appendix B) and qualify associated sample results as estimated (J) for detected results. If internal standard peak resolution is low (≤25%) then non-detects should qualified as unusable (R), otherwise, qualify all nondetects as estimated

(UJ) when recoveries are low. If more than two internal standards in any one sample exceed the criteria, contact the laboratory for clarification and document all subsequent discussions in the validation narrative.

7.9 COMPOUND IDENTIFICATION AND QUANTITATION

Criteria for compound identification and quantitation are established to reduce the possibility of false identification and quantitation of target compounds. Review the laboratory data for positive results against the following criteria:

- Review the chromatograms for interferences such as extraneous peaks or split peaks at the various monitoring masses where positive dioxin/furan congeners are detected. If the proper ions were not monitored, reject associated data (R).

- Specifically determine if polychlorinated diphenyl ether (PCDPE) interferences are present above a signal to noise ratio of 2.5 by reviewing the data against the list of the monitoring masses for PCDPE interferences provided in Method 8290. If interferences are noted for detected results, qualify the associated data as estimated (J for detects, UJ for non-detects).

- Verify that positive sample results exhibit simultaneous peak response for both the quantitation and confirmation ion masses; if not, qualify positive results as estimated (J).

- Verify that the signal to noise ratio for the quantitation ion is greater than or equal to 2.5; if not, qualify positive results as estimated (J).

- Check that chlorine isotope ratios are within the limits specified in Table 7-1; if not, discuss in the validation narrative.

- Review the reported retention times and check that positive results are within -1 to 3 seconds of the associated labeled internal standard at the quantitation mass, and are within the retention time windows established by the performance check solution. If retention times criteria are exceeded, reject the associated data (R).

7.9.1 Reported Quantitation Limits

Recalculate the results and verify compliance with the project specific detection limits. If the laboratory is unable to meet the detection limits within a factor of five and no known or suspected interferences are present in the sample, contact the laboratory for clarification and document all subsequent discussions in the validation narrative.

Table 7-1. Chlorine Isotope Ratio Control Limits

COMPOUND GROUP	CONTROL LIMITS
Tetra-CDD/CDF	0.65 to 0.89
Penta-CDD/CDF	1.32 to 1.78
Hexa-CDD/CDF	1.05 to 1.43
^{13}C-Hexa-CDD/CDF	0.43 to 0.59
Hepta-CDD/CDF	0.88 to 1.20
^{13}C-Hepta-CDD/CDF	0.37 to 0.51
Octa-CDD/CDF	0.76 to 1.02

(Source: EPA 1990)

7.10 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A) and qualify affected data on the data qualification summary (Appendix B). Prepare a brief narrative summary that addresses data acceptability as related to project work plan and QAPjP requirements and according to the procedures specified in Section 10.

8.0 INORGANIC DATA REVIEW REQUIREMENTS

This section presents specific review requirements for inorganic analyses. The analytical requirements for these analyses are contained in the CLP SOW (EPA, 1988b) and data review requirements for inorganics analysis as referenced in the EPA validation guidelines (Bleyler, 1988). The data review requirements specified herein may be applied to data produced using procedures described in SW-846 (EPA 1986).

8.1 DATA PACKAGE COMPLETENESS

After receipt of the data package and completion of records management activities detailed in Section 2, the reviewer shall organize the data package according to the order of deliverables specified in Section 1 of the data validation checklist (Appendix A, Form A-6). Missing data review items that the reviewer deems necessary for completion of the validation shall require the reviewer to contact the laboratory for submittal of the needed item. All contacts with the laboratory must be documented on the appropriate form (Appendix B, Form B-8).

8.2 HOLDING TIMES

Review the chain-of-custody forms and the analysis run log forms. All samples must be collected in the proper containers, properly preserved, digested or distilled and analyzed within the following holding times as established under 40 CFR Part 136. Holding times will be calculated from the date of collection to the date of analysis.

- Metals, 6 months; water samples shall be preserved to pH < 2
- Mercury, 28 days; water samples shall be preserved to pH < 2
- Cyanide, 14 days; water samples shall be preserved to pH > 12

Complete the holding time summary (Appendix B) and if holding times are exceeded, qualify all results as estimated (J for detects, UJ for non-detects). If holding times are greatly exceeded, the reviewer must use informed professional judgment to determine the reliability of the data. The expected bias would be low and the reviewer may determine that results less than the IDL are unusable (R).

8.3 INSTRUMENT PERFORMANCE AND CALIBRATIONS

Review the laboratory submitted raw data sheets, analysts notebook records and instrument run logs to ensure that the laboratory has calibrated the instruments as required by the CLP SOW, (EPA, 1988b). During review of the calibration data complete the calibration data summary form (Appendix B) noting those calibration analyses that exceed the calibration criteria and the affected samples.

8.3.1 ICP Calibration

Review the ICP raw data and calibration reports to verify the calibration of the ICP as follows.

8.3.1.1 Initial ICP Calibration

Instruments must be calibrated daily and each time instrument is set up. Using the raw data, verify that the instrument was calibrated daily (and each time the instrument was set up) using a blank and at least one standard to establish the analytical curve of the ICP. If the minimum number of standards were not used for calibration, or if the instrument was not calibrated daily and each time the instrument was set up, qualify the data as unusable (R).

8.3.1.2 Initial and Continuing Calibration Verification

Verify that the initial calibration verification (ICV) and continuing calibration verification (CCV) standards were analyzed at the required frequency and check for calculation errors on at least one ICV and CCV standard using the formula specified in the CLP/SOW (EPA 1988b). If the ICV or CCV falls outside the acceptance windows (90% to 110%), qualify the results according to the following table.

ICV,CCV %R	SAMPLE RESULT	QUALIFIER
75 to 89, 111 to 125	>IDL	J
111 to 125	<IDL	ACCEPTABLE/NO QUALIFIER
75 to 89	<IDL	UJ
<75	>IDL	R
>125	>IDL	R
>125	<IDL	ACCEPTABLE/NO QUALIFIER

8.3.2 ICP Interference Checks

Verify in the raw data that the ICP interference check solutions were run at the beginning and end of each sample analysis run or at a minimum of twice every 8 hours of an analysis run, whichever is more frequent. Verify that the results for the ICS solution AB analysis fall within the control limits of ±20% of the true value and check all calculations in at least one ICS analysis. Qualify results according to the following table for samples with concentrations of aluminum, calcium, iron and magnesium ≥ their respective levels in the ICS solution. In addition, an evaluation for false positives and false negatives should be conducted if results greater than the instrument detection limit (IDL) or >|IDL| are observed for analytes not present in the ICS. If samples contain analytes at levels comparable to the interferant levels in the ICS sample results, qualify sample results as estimated (J for detects, UJ for non-detects).

ICS %	SAMPLE RESULT	QUALIFIER
>120	<IDL	ACCEPTABLE/NO QUALIFIER
>120	>IDL	J
50 to 79	>IDL	J
50 to 79	<IDL	UJ
<50	ANY	R

8.3.3 Atomic Absorption Analysis (AA)

Review the graphite furnace AA (GFAA) and cold vapor AA (CVAA) raw data to verify that instrument calibration meets the following criteria.

8.3.3.1 Initial Calibration

Verify, using the raw data, that the instrument was calibrated daily and each time the instrument was set up. A blank and at least three standards (four standards for mercury), one of which must be at the CRQL, must be used in establishing the analytical curve. If the minimum number of standards were not used for calibration, or if the instrument was not calibrated daily and each time the instrument was set up, qualify the data as unusable (R). Recalculate the correlation coefficient (r) of the standard curves, verify linearity, and check that the r value is ≥ 0.995. If the r value is < 0.995, qualify all affected results as estimated (J for detects, UJ for non-detects).

8.3.3.2 Initial and Continuing Calibration Verification

Verify that the ICV and CCV standards were analyzed within the required frequency; check calculations and verify reported values. Qualify data according to the following table.

ICV,CCV %R	SAMPLE RESULT	QUALIFIER
FURNACE AA		
75 to 89, 111 to 125	>IDL	J
111 to 125	<IDL	ACCEPTABLE/NO QUALIFIER
75 to 89	<IDL	UJ
<75	>IDL	R
>125	>IDL	R
>125	<IDL	ACCEPTABLE/NO QUALIFIER
MERCURY		
65 to 79, 121 to 135	>IDL	J
121 to 135	<IDL	ACCEPTABLE/NO QUALIFIER
65 to 79	<IDL	UJ
<65	>IDL	R
>135	>IDL	R
>135	<IDL	ACCEPTABLE/NO QUALIFIER

8.3.4 Cyanide Analysis

Review the cyanide data to verify that the following criteria were met during calibration.

8.3.4.1 Initial Calibration

Verify from the raw data, that the instrument was calibrated daily and each time the instrument was set up. A blank and at least three standards must be used in establishing the analytical curve. If the minimum number of standards were not used for calibration, or if the instrument was not calibrated daily and each time the instrument was set up, qualify the data as unusable (R). Check the distillation log to ensure that a mid-range standard was distilled, and if not, qualify all associated results as estimated (J). Calculate the correlation coefficient (r) of the standard curve(s) to verify linearity and that the r value was ≥ 0.995 for the photometric determination. If the r value was < 0.995, qualify all results as estimated (J for detects, UJ for non-detects).

8.3.4.2 Initial and Continuing Calibration Verification

Verify that the ICV and CCV standards were analyzed at the required frequency and check calculations and verify reported values. Qualify data according to the following table.

ICV,CCV %R	SAMPLE RESULT	QUALIFIER
70 to 84, 116 to 130	>IDL	J
116 to 130	<IDL	ACCEPTABLE/NO QUALIFIER
70 to 84	<IDL	UJ
<70	>IDL	R
>130	>IDL	R
>130	<IDL	ACCEPTABLE/NO QUALIFIER

8.4 BLANKS

Review the laboratory result reports, instrument raw data sheets and analyst notebook sheets. Blanks are analyzed as a means of determining contamination introduced by the laboratory or sampling operations. No contaminants should be present in the blanks. Contamination may be introduced from the sample handling and processing, sample containers and field sampling procedures and equipment. The blank analyses may not involve the same weights, volumes or dilution factors as the associated samples since soil samples are reported in $\mu g/Kg$ units and the associated blanks except for the preparation blank, are reported in $\mu g/L$ units. It may be easier to work from the raw data when reviewing the blank data.

Prior to reviewing the blanks, complete the blank data summary form summarizing the detected results in all the blanks.

8.4.1 Laboratory Blanks

Review the reported results for the laboratory blanks and the raw data and verify that results were accurately reported. Verify the laboratory has analyzed one preparation blank with each batch of samples and matrices and has analyzed initial calibration blank (ICB) and continuing calibration blank (CCB) samples at the correct frequency as specified in the CLP SOW (EPA 1988b). For any blank with an analyte concentration > IDL but < CRQL, qualify as non-detects (U) associated samples with concentrations of the analyte < five times the highest blank concentration.

8.4.2 Field Blanks

Following the review of laboratory blanks, review the field sampling documentation to identify the field blank samples and sample types. Review the results for the field blanks for target analytes greater than the IDL, and if present, note in the validation narrative. Samples that contain less than five times the highest valid field blank concentration are qualified as non-detects (U) at the reported sample concentration.

8.5 ACCURACY

Laboratory performance and compliance with project specific and analytical accuracy requirements is determined by a review of matrix spike, laboratory control, and performance audit sample recovery. The laboratory should conduct at least one matrix spike and laboratory control sample analysis on each matrix for each SDG or every 20 samples whichever is more frequent.

8.5.1 Matrix Spike Recovery

The matrix spike sample analysis provides information about the effect of each sample matrix on the digestion and measurement methodology. Review the spike sample recovery results and verify that results are within the limits of 75% to 125% recovery unless sample concentration exceeds the spike concentration by a factor of four or more. Recalculate all matrix spike samples using the formula listed in the CLP SOW (EPA 1988b).

Qualify the sample results based on the following table.

SPIKE RECOVERY	SAMPLE RESULT	QUALIFIER
>125%	<IDL	ACCEPTABLE/NO QUALIFIER
>125% or <75%	>IDL	J
30 - 74%	<IDL	UJ
<30%	<IDL	R

8.5.2 Laboratory Control Sample Recovery

The laboratory control sample (LCS) serves as a monitor of the overall performance of all steps in the analysis, including the sample preparation. Review the reported results against the raw data, check all calculations and verify the recoveries fall within the control limits of 80-120% for the aqueous LCS for all analytes (except antimony and silver) and within the published control limits for the solid LCS. If the LCS recovery falls outside the control limits qualify the data according to the following table.

LCS PERCENT RECOVERY	SAMPLE RESULT	QUALIFIER
AQUEOUS MATRIX		
50% - 79%, >120% >120% 50% - 79% <50%	>IDL <IDL <IDL ANY	J ACCEPTABLE/NO QUALIFIER UJ R
SOLID MATRIX		
< OR > CONTROL LIMIT < CONTROL LIMIT > CONTROL LIMIT	>IDL <IDL <IDL	J UJ ACCEPTABLE/NO QUALIFIER

8.5.3 Performance Audit Analyses

Contact the WHC project coordinator for the identity, source and control limits for any performance audit sample submitted with the sample group. If the results for any analyte are outside the control limits contact the laboratory for explanation and reanalysis if required by the work plan and QAPjP.

8.6 PRECISION

The review of field and laboratory precision provides information on the laboratory reproducibility and whether sampling activities are adequate to acquire consistent samples.

8.6.1 Laboratory Duplicate Samples

Review the raw data and duplicate report to verify that results fall within the control limits. Verify that the laboratory has performed one duplicate sample analysis on each matrix for each SDG or 20 samples whichever is greater; check all calculations using the formula provided in the CLP SOW (EPA, 1988b). Qualify sample results according to the following table.

RPD	SAMPLE RESULT	QUALIFIER
>20 (>35 soils) ±CRQL or (±2xCRQL soils)	>5X CRQL <5X CRQL	J J

8.6.2 ICP Serial Dilution

The ICP serial dilution is used to determine whether significant physical or chemical interferences exist due to sample matrix. Check the raw data and recalculate all of the %D between the initial and diluted results of analytes for which the sample concentration is ≥ 50 times the IDL, to verify that the dilution analysis results agree with the reported results. Check the raw data for evidence of negative interference, diluted sample results that are significantly higher than the original sample. If sample concentration is ≥ 50 times the IDL for an analyte and the %D is outside the control limits of ±10%, qualify the associated data as estimated (J). If negative interference is suspected, qualify the results using informed professional judgement and discuss the qualification in the validation narrative.

8.6.3 Field Duplicate Analysis

Contact the WHC project manager for the identity of the primary and field duplicate samples. Complete the precision data summary, calculate the RPD values and note the results in the validation narrative.

8.6.4 Field Split Samples

Contact the WHC project manager for the identity of the primary and field split samples. Complete the precision data summary, calculate the RPD values and note the results in the validation narrative.

8.7 FURNACE AA QUALITY CONTROL

Duplicate injections and furnace post digestion spikes establish the precision and accuracy of the individual GFAA determinations.

8.7.1 Duplicate Injections

Check the raw data to verify that the GFAA sample analysis included duplicate injections for each sample, standard and blank. Verify that the RSD for the duplicate injection results are within the control limits of ±20% for samples with concentrations > CRQL. If the duplicate injections are outside the limits, and the sample has not been reanalyzed or the reanalysis is out specification, qualify the associated data as estimated (J for detects, UJ for non-detects).

8.7.2 Analytical Spike Recovery

Review the furnace AA data and verify that analytical spikes were conducted and that percent recoveries are of ≥85% and ≤115%. Qualify results according to the following table.

ANALYTICAL SPIKE RECOVERY	SAMPLE RESULT	QUALIFIER
<40%	>IDL	J
≥10% and <40%	<IDL	UJ
<10%	<IDL	R
SAMPLE ABSORBANCE <50% OF ANALYTICAL SPIKE ABSORBANCE		
<85% or >115%	>IDL	J
<85% or >115%	<IDL	UJ

If sample absorbance is >50% of the analytical spike absorbance and the %R is outside the control limits the laboratory is required to analyze the sample by Method of Standard Additions (MSA). If a sample required MSA analysis but was not analyzed, qualify the data as estimated (J). If any of the samples analyzed by MSA were not spiked at the appropriate levels (50%, 100% and 150% of the sample concentration), qualify the data as estimated (J). If the MSA correlation coefficient is <0.995, qualify the data as estimated (J).

8.8 ANALYTE QUANTITATION AND DETECTION LIMITS

Examine the raw data to verify the correct calculation of at least 20% of the sample results reported by the laboratory. Check calculations using the formula provided in the CLP SOW (EPA 1988b). Examine the raw data for any anomalies such as baseline drifts, negative absorbance, omissions, and legibility. Verify there are no transcription or reduction errors and check to ensure that results reported for the ICP fall within the linear range of the instrument. For non-ICP parameters check that results fall within the calibrated range. In addition check that instrument detection limits were below the CRQL levels as required by the CLP SOW (EPA 1988b).

8.9 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A), prepare a brief narrative summary of the data acceptability and quality control deficiencies, qualify affected data as determined from the review, and summarize the qualified results as specified in Section 10.

9.0 WET CHEMISTRY DATA REVIEW REQUIREMENTS

This section presents specific review requirements for wet chemistry analyses. Table 9-1 provides a list of the specific analytical parameters and applicable reference methods. Data review requirements are based upon the reference methods and where applicable, on the EPA data validation guidance (Bleyler 1988).

Successful completion of the wet chemistry data review will require the reviewer to have the following references available:

- Current approved versions of the project-specific technical work plan and QA Project Plan,
- The applicable current approved contract laboratory QA Project Plan and standard operating procedures (SOPs), and
- Copies of the analytical reference methods as listed in Table 9-1.

9.1 DATA PACKAGE COMPLETENESS

After receipt of the data package and completion of records management activities detailed in section 2, the reviewer shall organize the data package according to the order of deliverables specified in section 1 of the data validation checklist (Appendix A, Form A-7). Observation of missing data review items that the reviewer deems necessary for completion of the validation shall prompt the reviewer to contact the laboratory for submittal of the needed item. All contacts with the laboratory must be documented on the appropriate form (Appendix B, Form B-8).

9.2 HOLDING TIMES

Review the chain-of-custody forms and the raw data. All water samples must be collected in the proper containers, properly preserved and analyzed within the holding times as established under 40 CFR Part 136 and as listed in Table 9-1. The holding time summary (Appendix B) must be completed and appended to the checklist. If the holding times are not met, qualify all sample data as estimated (J for detects, UJ for non-detects).

Table 9-1. Wet Chemistry Analysis Parameters

ANALYTICAL PARAMETER	METHOD OF ANALYSIS	HOLDING TIME, DAYS	ACCURACY % RECOVERY	PRECISION RPD	CRQL[5]
pH	EPA 150.1[1] or APHA 423[2]	N/A	N/A	20	0.05 su
Specific conductance	EPA 120.1[1] or APHA 205[2]	28	75-125	20	5 umhos
Total Dissolved Solids (180°C)	EPA 160.2[1] or APHA 209B[2]	7	75-125	20	10
Nitrate+nitrite as N	EPA 300.0[3] or EPA 353.2[1]	28	75-125	20	0.1
Fluoride	EPA 300.0[3] or EPA 340.2[1]	28	75-125	20	0.1
Sulfate	EPA 300.0[3] or EPA 375.4[1]	28	75-125	20	0.1
Chloride	EPA 300.0[3] or EPA 325.3[1]	28	75-125	20	0.1
Bromide	EPA 300.0[3] or EPA 320.1[1]	28	75-125	20	0.1
Ortho-phosphate (as P)	EPA 300.0[3] or EPA 365.2[1]	2	75-125	20	0.1
Alkalinity, total as $CaCO_3$	EPA 310.1[1] or APHA 403[2]	14	75-125	20	0.1
Ammonia as N	EPA 350.3[1]	28	75-125	20	0.1
Chemical Oxygen Demand	EPA 410.1[1] or APHA 508A[2]	28	75-125	20	10
Total Organic Carbon	EPA 415.1[1] or APHA 505[2]	28	75-125	20	2.0
Total Organic Halogen	EPA 9020[4]	7	75-125	20	0.05

[1]Method from Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, U.S. Environmental Protection Agency, Washington, D.C.

[2]Method from Standard Methods for the Examination of Water and Wastewater, 15th Edition, American Public Health Association, Washington, D.C.

[3]Method from The Determination of Inorganic Anions in Water by Ion Chromatography, Method 300.0, EPA-600/4-84-017, U.S. Environmental Protection Agency, Washington, D.C.

[4]Method from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition, U.S. Environmental Protection Agency, Washington, D.C.

[5]CRQL values are mg/L units except where noted.

9.3 CALIBRATIONS

Review the methods, laboratory submitted raw data sheets, analyst notebook records and instrument run logs (if applicable) to ensure that the laboratory has calibrated the instruments and other ancillary equipment as required by the approved laboratory SOP.

9.3.1 Initial calibration

Check the results and raw data for each analytical parameter to verify that the following calibration procedures were conducted prior to the analysis of samples:

- At least two reference buffers or standards were used to calibrate the pH and conductivity meters;
- an analytical balance check was conducted prior to analysis of TDS samples;
- at least a blank and three standards were used to establish the ion chromatography, ion selective electrode, spectrophotometer, TOC analyzer and TOX analyzer calibration prior to sample analysis and the correlation was $\geq$0.995; and
- the titrant normality for alkalinity analysis was checked.

If the calibration requirements were not met, contact the laboratory for clarification and, if necessary, qualify affected data as unusable (R). Document all discussions with the laboratory on the validation report narrative.

9.3.2 Initial and Continuing Calibration Verification

Verify that the ICV and CCV standards were analyzed (for all analyses except TDS) with the required frequency or every 20 samples and check calculations. Complete the calibration data summary form and if the ICV or CCV percent recovery falls outside the acceptance windows of 90 to 110% (75 to 125% for COD, TOC & TOX) qualify associated data as estimated (J for detects, UJ for non-detects).

9.4 BLANKS

Review the laboratory result reports, instrument raw data sheets and analyst notebook sheets. Blanks are analyzed as a means of determining contamination introduced by the laboratory or sampling operations. Contamination may be introduced from sample handling and processing activities, sample containers, and field sampling procedures and

equipment. Note that the blank analyses may not involve the same weights, volumes or dilution factors as the associated samples. In particular, soil sample results are reported in µg/Kg units and the associated blank samples (with the possible exception of the preparation blank) are reported in µg/L units. It may be easier to work from the raw data when reviewing the blank results.

Prior to beginning the review of blank data, complete the blank and sample data summary (Appendix B) listing all detected analytes in the blanks and samples.

9.4.1 Laboratory Blanks

Review the results for laboratory blanks and the raw data; verify the laboratory has analyzed one preparation blank with each sample batch and each matrix and has analyzed ICB and CCB samples at the required frequency (except for TDS). Check that all blank results are less than the CRQL and, if not, qualify associated data less than five times the amount found in the blank as undetected (U).

9.4.2 Field Blanks

Following the review of laboratory blanks, review the chain of custody and/or sample analysis request documentation to identify the field blank samples and sample types. Review the results for the field blanks; if the field blank(s) contain target parameters greater than the MDL, discuss in the validation report narrative. Samples that contain less than five times the highest valid field blank concentration are qualified as undetected (U) at the reported sample concentration.

9.5 ACCURACY

Laboratory performance and compliance with project specific and analytical accuracy requirements is determined by a review of matrix spike, laboratory control and performance audit sample recovery. The laboratory should conduct at least one LCS and one MS (TDS, IC, and ISE analyses) or MS/MSD (TOC and TOX analyses) analysis on each applicable matrix for each SDG, or every 20 samples, whichever is more frequent.

9.5.1 Matrix Spike Recovery

The matrix spike sample analysis provides information about the effect of each sample matrix on the digestion and measurement methodology. Review spike reports, verify that the results fall within the limits of 75% to 125% unless sample concentration exceeds the spike concentration by a factor of 4 or more. Check calculations and complete the accuracy data summary form (Appendix B). If the spike recovery is outside the control limits and the sample results are > CRQL, qualify the data as estimated (J). If the spike

recovery is < 30% and the sample results are < IDL, qualify the data for the associated samples as unusable (R) and contact the laboratory for clarification; document all subsequent discussions in the validation narrative. Use informed professional judgment if the laboratory used a field blank for the matrix spike analysis.

9.5.2 Laboratory Control Sample Recovery

The laboratory control sample serves as a monitor of the overall performance of all steps in the analysis, including the sample preparation. Review the report forms and raw data, verify results for the LCS for applicable methods, and check calculations. Check that recoveries are within the control limits of 80% to 120% for the aqueous LCS and within the established control limits for the solid LCS. If the LCS recovery falls outside the control limits qualify the data as follows:

LABORATORY CONTROL SAMPLE PERCENT RECOVERY	SAMPLE RESULT	QUALIFIER
AQUEOUS MATRIX		
50 to 79%, > 120%	> IDL	J
> 120%	< IDL	ACCEPTABLE/NO QUALIFIER
50 to 79%	< IDL	UJ
< 50%	ANY	R
SOLID MATRIX		
<> CONTROL LIMIT	> IDL	J
< CONTROL LIMIT	< IDL	UJ
> CONTROL LIMIT	< IDL	ACCEPTABLE/NO QUALIFIER

9.5.3 Performance Audit Analyses

The reviewer shall contact the WHC project coordinator for the performance audit sample number and the associated control limits. Complete the accuracy data summary form noting the analyses that exceed the control limits. If the results for any parameter are outside the control limits contact the laboratory for clarification and document all subsequent discussions in the validation narrative.

9.6 PRECISION

The review of field and laboratory precision provides information on the reproducibility of laboratory analysis and whether sampling activities are adequate to acquire consistent samples.

9.6.1 Laboratory Duplicates

Review the raw data and report forms; verify that results fall within the control limits and check all calculations. Check that the laboratory has performed one duplicate sample analysis for each SDG or 20 samples whichever is greater. Complete the precision data summary form (Appendix B) and qualify all data as estimated (J) when the RPD is greater than 20% (35% for soils) for sample results greater than five times the CRQL, or ± the CRQL (±2x the CRQL for soils), when sample results are less than five times the CRQL.

9.6.2 Field Duplicates

The reviewer shall contact the WHC project coordinator for the sample numbers of the primary and field duplicate samples. Check the raw data and reports to verify that the reported results are correct. Complete the precision data summary form (Appendix B) and calculate the RPD values. Note the results of the field duplicates evaluation in the validation narrative.

9.6.3 Field Split Sample Analyses

The reviewer shall contact the WHC project coordinator for the sample numbers of the primary and field split samples. Check the raw data and reports to verify that the reported results are correct. Complete the precision data summary form (Appendix B), calculate the RPD, values and note the results of the field split sample evaluation in the validation narrative.

9.7 ANALYTE QUANTITATION AND DETECTION LIMITS

Examine the raw data to verify the correct calculation of at least 20% of the sample results reported by the laboratory. Raw data shall be compared to the reported results and examined for anomalies, transcription or reduction errors. Check that all sample results reported were within the calibrated range of the instrument and verify that instrument detection limits were below CRQL values.

9.8 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A), and prepare a brief narrative summary of the data acceptability and any observed quality control deficiencies. Qualify affected data as determined from the review and summarize the requalified results as specified in Section 10.

10.0 REPORTING REQUIREMENTS

This section presents reporting requirements for validation reports on both a sample group and overall case basis, where several groups of sample analyses are summarized for inclusion into individual environmental site investigation reports. All validation reports must be completed and transmitted to the WHC project coordinator within 21 days of receipt of the complete laboratory data package.

10.1 VALIDATION REPORTS

After completing the data review for a specific analysis type or group of analyses, complete the appropriate checklist and summarize the results of the data review in a narrative summary that addresses any quality control deficiencies identified, and their effect on the data quality; attach copies of the checklists, laboratory reports, QC reports and other supporting documentation and forward to the WHC project coordinator with a summary of the validated data in written and electronic format (Section 10.2).

The validation narrative should address at a minimum the following elements as they are related to the data quality objectives of the project and shall be provided to the WHC project coordinator for review in the format of a technical memorandum addressing the following items:

- Introduction and Summary - This section of the memorandum shall provide a short introduction of the sample types, analyses conducted, laboratories involved, and applicable plans and specifications.

- Data Quality Objectives - This section of the report shall provide a summary of the degree to which the project specific data quality objectives were met as related to the sample analyses. Separate narrative summaries of the precision, accuracy, representativeness, completeness and comparability of the data reviewed shall be addressed, where applicable.

- Qualified Data - This section of the report shall provide a tabulated summary of the qualified data or hardcopy printout of the qualified electronic data. This summary may include copies of the laboratory sample concentration reports, QC summaries and other applicable documentation submitted to the laboratory. At a minimum the tabular summary must provide the sample number, sample collection date, sample location, sample type, constituent name, constituent result, result qualifier and constituent reporting units. In preparation of the tabulated data summary, the reviewer must have a system of performing a 100% check for transcription errors of all data against the written documentation; procedures shall be submitted to the WHC project coordinator for approval prior to use.

- Conclusions - This section of the report shall provide a summary of the results and the discussion of any QA/QC deficiencies that affect the usability of the data as related to the project specifications and requirements.

The completed validation narrative, with all supporting calculation, checklists, and raw data shall be reviewed by the subcontractor's QA Officer for compliance with the requirements at this CLP SOW prior to submittal to the WHC project coordinator.

At the completion of a project that may involve several sample analysis groups, a final narrative summary will be completed, reviewed, and submitted to the project coordinator using the format and content requirements as specified above. An example of a typical validation narrative report and data summary for organic analysis data is provided in Appendix C.

10.2 ELECTRONIC DATA TRANSMITTAL REQUIREMENTS

Results of the validated data are to be provided in the format described in Table 10-1 on 5.25-inch flexible disk in MS-DOS[1] low density or high density format compatible with the applicable subject areas specified in the Hanford Environmental Information System (HEIS) Users Manual (WHC 1990). Each record in the file is designed to contain the analytical results for one chemical analysis parameter and all fields in the record are to be fixed-length, containing no special format codes, delimiters or separators. Data entry fields marked with an asterisk (*) in Table 10-1 refer to fields in the transmittal file that must contain the specified information and may make up part of the unique identifier assigned by HEIS for retrieval of the record. The reviewer must have a system in place for verifying the accuracy of the electronic data with the written record if changes to the result qualifiers are made as a result of the validation effort; procedure shall be submitted to the WHC project coordinator for approval prior to use. At a minimum a 100% check of all data against the written documentation must be performed.

The WHC project coordinator may specify options for electronic data submittals on a case by case basis since laboratory electronic data transmittal formats are currently in development.

[1]MS-DOS is a trademark of Microsoft Corporation, Redmond, Washington.

Table 10-1. Electronic Data Transmittal Format

FIELD NAME	FIELD LENGTH	RECORD POSITION	DESCRIPTION
samp_num (*)	12	1 - 12	The unique number assigned by HEIS to identify the sample.
samp_date (*)	8	13 - 20	The sample collection date in the format: MM/DD/YY
samp_time	5	21 - 25	The sample collection time in the format: HH:MM using a 24h clock.
location	15	26 - 40	The location where the sample was collected such as the well name, borehole location, or sample location.
media	3	41 - 43	The sample media code as specified by HEIS in the format: AT = atmospheric BI = biota GS = geologic soil GW = ground-water Q = sample blank SG = soil gas SS = surface soil SW = surface water
samp_qual	2	44 - 45	The sample type code as specified by HEIS in the format: BB = bottle blank BS = blind standard EB = equipment blank ES = equipment spike FB = field blank FS = field spike PB = transport blank TB = trip blank TS = trip spike XB = transfer blank

Table 10-1., (Cont.) Electronic Data Transmittal Format

FIELD NAME	FIELD LENGTH	RECORD POSITION	DESCRIPTION
drsc_samp_type	1	46	The QC sample type as specified by HEIS in the format: D = duplicate R = replicate S = split C = composite
con_id (*)	10	47 - 56	The unique identifier of the chemical constituent parameter, the Chemical Abstracts Services (CAS) number, or an identifier assigned by HEIS.
con_long_name (*)	40	57 - 96	The name of the chemical constituent parameter.
value_rptd (*)	12	97 - 108	The chemical analysis result in scientific format such as 1.26E-4
qualifier (*)	6	107 - 114	The result qualifier assigned by the laboratory and/or validation as specified in the CLP/SOW and the validation statement of work.
counting_error (*)	12	115 - 126	The 2-sigma counting error reported by the laboratory for radiochemical analyses.
retention_time (*)	8	127 - 134	The chromatographic retention time for the compound reported if the compound is a TIC.
units_std (*)	5	135 - 139	The reporting units of the result reported in the field value_rptd.

(*) - Indicates the field is required for transmittal of the data and may be used by HEIS to make up the unique identifier for retrieval and display of the record information.

11.0 REFERENCES

APHA 1985, Standard Methods for the Examination of Water and Wastewater, 16th Edition, American Public Health Associates, Washington, D.C.

Bleyler, R., 1988a, Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses, United States Environmental Protection Agency, Hazardous Site Evaluation Division, Washington, D.C.

Bleyler, R., 1988b, Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses, United States Environmental Protection Agency, Hazardous Site Evaluation Division, Washington, D.C.

EPA 1977, Handbook for Analytical Quality Control in Radioanalytical Laboratories, EPA-600/4-77-088, United States Environmental Protection Agency, Office of Research and Development, Washington, D.C.

EPA 1979, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, United States Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

EPA 1980, Prescribed Procedures for the Measurement of Radioactivity in Drinking Water, United States Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

EPA 1986, Test Methods for Evaluating Solid Waste (SW-846), Third Edition; United States Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

EPA 1988a, USEPA Contract Laboratory Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, United States Environmental Protection Agency, Washington D.C.

EPA 1988b, USEPA Contract Laboratory Program, Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration, United States Environmental Protection Agency, Washington, D.C.

EPA 1989a, Method 8290, Determination of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans by High-Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS), Revision 0, October 1989, United States Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C.

Harley, 1972, EML Procedures Manual, HASL-300, Environmental Measurement Laboratory, United States Department of Energy, New York, NY.

WHC 1990, Westinghouse Hanford Company Hanford Environmental Information System (HEIS) User's Manual, WHC-EP-0372, Volume 1, Westinghouse Hanford Company, Richland, Washington.

APPENDIX A

DATA VALIDATION CHECKLISTS

VOLATILE ORGANIC DATA REVIEW CHECKLIST - FORM A-1

PROJECT:	REVIEWER:	DATE:
LABORATORY:	CASE:	SDG:
SAMPLES/MATRIX:		

1. DATA PACKAGE COMPLETENESS

Review the data package for completeness and check off the items below. If any data review elements are missing contact the laboratory for submittal.

Data Package Item	Present?:	Yes	No	N/A
Case Narrative		—	—	—
Data Summary		—	—	—
Chain-of-Custody		—	—	—
QC Summary				
Surrogate report		—	—	—
MS/MSD report		—	—	—
Blank summary report		—	—	—
GC/MS tuning report		—	—	—
Internal standard summary report		—	—	—
Sample Data				
Sample reports		—	—	—
TIC reports for each sample		—	—	—
RIC reports for all samples		—	—	—
Raw and corrected spectra for all detected results		—	—	—
Raw and corrected library search data for all reported TIC		—	—	—
Quantitation and calculation data for all TIC		—	—	—
Standards Data				
Initial calibration report		—	—	—
RIC and quantitation reports for initial calibration		—	—	—
Continuing calibration reports		—	—	—
RIC and quantitation reports for cont. calibrations		—	—	—
Internal standard summary report		—	—	—

Data Package Item	Present?:	Yes	No	N/A
Raw QC Data				
Tuning report, spectra and mass lists		___	___	___
Blank analysis reports		___	___	___
TIC reports for all blanks		___	___	___
RIC and quantitation reports for blanks		___	___	___
Raw and corrected spectra for all detected results in blanks		___	___	___
Raw and corrected library search data for all reported TIC		___	___	___
Quantitation and calculation data for all TIC		___	___	___
MS/MSD report forms		___	___	___
RIC and quantitation reports for MS/MSD		___	___	___
Additional Data				
Moisture/% solids data sheets		___	___	___
Reduction formulae		___	___	___
Instrument time logs		___	___	___
Chemist notebook pages		___	___	___
Sample preparation sheets		___	___	___

2. HOLDING TIMES

Complete the holding time summary form listing all samples and dates of collection and analysis.

Were all samples analyzed within holding time? Yes No N/A

ACTION: If any holding times were exceeded, qualify associated samples as estimated (J for detects or UJ for non-detects).

3. INSTRUMENT CALIBRATION AND TUNING

3.1 GC/MS TUNING

Is a BFB tune report present for each applicable 12h period? Yes No N/A

Do all tunes on all instruments meet the tuning criteria? Yes No N/A

Do all tunes on all instruments meet the expanded criteria? Yes No N/A

Has the laboratory made any calculation or transciption errors? Yes No N/A

Have the proper significant figures been reported? Yes No N/A

ACTION: If the mass calibration is out of specification but within the expanded criteria, qualify associated data as estimated (J for detects or UJ for non-detects). If all tuning criteria are missed, qualify all associated data as unusable (R).

3.2 INITIAL CALIBRATION

Is an initial calibration report provided for all instruments?	Yes	No	N/A
Are all RSD values ≤30%?	Yes	No	N/A
Are all RRF values ≥0.05?	Yes	No	N/A

ACTION: If any RRF value is out of specification qualify all detected results for the particular compound as estimated (J) and all non-detects as unusable (R). If any RSD value is out of specification qualify all associated data as estimated (J for detects or UJ for non-detects).

3.3. CONTINUING CALIBRATION

Is a continuing calibration report present for all 12h periods in which associated samples were analyzed ?	Yes	No	N/A
Are all RRF values ≥0.05?	Yes	No	N/A
Are all %D values ≤25%?	Yes	No	N/A

ACTION: If any RRF value is out of specification qualify all associated detected results as estimated and all non-detects as unusable (R). If any %D is out of specification, qualify all associated results as estimated (J for detects or UJ for non-detects).

4. BLANKS

4.1 LABORATORY BLANKS

Has the laboratory conducted a method blank analysis per matrix for every 12h period in which samples were analyzed?	Yes	No	N/A
Are TCL compounds present in the laboratory blanks?	Yes	No	N/A

ACTION: Qualify all sample results <10X the highest blank concentration for the common laboratory contaminants, as non-detects (U) or at the SQL if the result is < CRQL. Qualify all remaining sample results <5X the blank concentration in similar fashion.

4.2. FIELD BLANKS

Are TCL compounds present in the field blanks? Yes No N/A

ACTION: Qualify all detected sample results less than or equal to five times the amount in any valid field blank as non-detects (U) and note the field blank results in the validation narrative.

5. ACCURACY

5.1 SURROGATE RECOVERY

Are any surrogate recoveries out of specification? Yes No N/A

Are any surrogate recoveries less than 10%? Yes No N/A

Are any method blank surrogate recoveries out of specification? Yes No N/A

ACTION: Qualify all associated sample results as estimated (J for detects or UJ for non-detects) for surrogates out of specification but greater than 10%. Qualify all associated positive sample results as estimated (J) and all non-detect results as unusable (R) for all surrogates below 10%. If method blank surrogates are out of specification and the associated sample surrogates are acceptable no qualification is necessary, however, the laboratory should be contacted for an explanation.

5.2 MATRIX SPIKE RECOVERY

Has an MS/MSD analysis been conducted per matrix in the sample group? Yes No N/A

Are MS/MSD recoveries within specification? Yes No N/A

Are there any calculation errors? Yes No N/A

ACTION: If an MS/MSD analysis has not been conducted contact the laboratory for an explanation. Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the results in the validation narrative. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

5.3 PERFORMANCE AUDIT SAMPLES

Are the performance audit sample results within the acceptance limits? Yes No N/A

ACTION: Note the results of the performance audit sample in the validation narrative.

6. PRECISION

6.1 MATRIX SPIKE/MATRIX SPIKE DUPLICATES

Are RPD values within specification? Yes No N/A

Are there any calculation errors? Yes No N/A

ACTION: Review the MS/MSD results in conjunction with other QC data such as field duplicates and not the results in the validation narrative. If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

6.2 FIELD DUPLICATE SAMPLES

Are field duplicate RPD values acceptable? Yes No N/A

ACTION: Note the results of the field duplicate samples in the validation narrative.

6.3 FIELD SPLIT SAMPLES

Are field split RPD values acceptable? Yes No N/A

ACTION: Note the results of the field split samples in the validation narrative.

7. SYSTEM PERFORMANCE

7.1 INTERNAL STANDARDS PERFORMANCE

Are any internal standard area counts outside the acceptance limits? Yes No N/A

Are retention times for any internal standard outside the ±30 second windows established by the most recent calibration check? Yes No N/A

ACTION: If the area counts are outside the acceptance limits qualify all associated results as estimated (J for detects or UJ for non-detects). If area counts are outside the acceptance limits and the retention time criteria are not met qualify all non-detects in the associated samples as unusable (R).

8. COMPOUND IDENTIFICATION AND QUANTITATION

8.1 COMPOUND IDENTIFICATION

Are detected compounds within ±0.06 relative retention time units of the associated calibration standard? Yes No N/A

Are all ions at a relative intensity of ≥10% in the standard spectra present in the sample spectra? Yes No N/A

Do the relative intensities between the standard and sample spectra agree within 20%? Yes No N/A

Have all ions >10% in the sample spectra that are not present in the standard spectra been explained? Yes No N/A

ACTION: If compound identification is in error and retention time and mass spectral criteria are exceeded qualify all affected positive results as unusable (R). If cross-contamination between analyses is suspected, qualify affected data as unusable (R). Note the results in the validation narrative.

8.2 REPORTED RESULTS AND QUANTITATION LIMITS

Has the laboratory used the correct RRF values and internal standard(s) for quantitation? Yes No N/A

Are results and quantitation limits calculated properly? Yes No N/A

Has the laboratory reported the sample quantitation limits within five times the CRQL values? Yes No N/A

ACTION: If the results and quantitation limits are in error contact the laboratory for clarification and note in the validation narrative.

8.3 TENTATIVELY IDENTIFIED COMPOUNDS (TIC)

Has the laboratory conducted a spectral library search on all candidate TIC peaks in accordance with the analytical SOW? Yes No N/A

Has the laboratory properly identified and coded all TIC? Yes No N/A

ACTION: If the laboratory has failed to search the minimum number of TIC peaks in the chromatogram contact the laboratory for submittal of the required data. Qualify as non-detects (U) all TIC compounds present in samples and blanks using the review criteria specified in the validation requirements. If TIC identification is in error sample results should be qualified as non-detects (U) or unusable (R). If TIC identifications are judged valid, qualify the results as presumptive and estimated (JN).

9. OVERALL ASSESSMENT AND SUMMARY

Has the laboratory conducted the analysis in accordance with the analytical SOW?	Yes	No	N/A
Were project specific data quality objectives met for this analysis?	Yes	No	N/A

ACTION: Summarize all the data qualifications recommended in the foregoing sections, and complete the data validation narrative according to the requirements of Section 10 of the data validation requirements.

COMMENTS (attach additional sheets as necessary):

SEMI-VOLATILE ORGANIC DATA REVIEW CHECKLIST - FORM A-2

PROJECT:	REVIEWER:	DATE:
LABORATORY:	CASE:	SDG:
SAMPLES/MATRIX:		

1. DATA PACKAGE COMPLETENESS

Review the data package for completeness and check off the items below. If any data review elements are missing contact the laboratory for submittal.

Data Package Item	Present?:	Yes	No	N/A
Case Narrative		___	___	___
Data Summary		___	___	___
Chain-of-Custody		___	___	___
QC Summary				
Surrogate report		___	___	___
MS/MSD report		___	___	___
Blank summary report		___	___	___
GC/MS tuning report		___	___	___
Internal standard summary report		___	___	___
Sample Data				
Sample reports		___	___	___
TIC reports for each sample		___	___	___
RIC reports for all samples		___	___	___
Raw and corrected spectra for all detected results		___	___	___
Raw and corrected library search data for all reported TIC		___	___	___
Quantitation and calculation data for all TIC		___	___	___
Standards Data				
Initial calibration report		___	___	___
RIC and quantitation reports for initial calibration		___	___	___
Continuing calibration reports		___	___	___
RIC and quantitation reports for cont. calibrations		___	___	___
Internal standard summary report		___	___	___

Data Package Item	Present?:	Yes	No	N/A
Raw QC Data				
Tuning report, spectra and mass lists		___	___	___
Blank analysis reports		___	___	___
TIC reports for all blanks		___	___	___
RIC and quantitation reports for blanks		___	___	___
Raw and corrected spectra for all detected results in blanks		___	___	___
Raw and corrected library search data for all reported TIC		___	___	___
Quantitation and calculation data for all TIC		___	___	___
MS/MSD report forms		___	___	___
RIC and quantitation reports for MS/MSD		___	___	___
Additional Data				
Moisture/% solids data sheets		___	___	___
Reduction formulae		___	___	___
Instrument time logs		___	___	___
Chemist notebook pages		___	___	___
Sample preparation sheets		___	___	___

2. HOLDING TIMES

Were all samples extracted within holding time?	Yes	No	N/A
Were all samples analyzed within holding time?	Yes	No	N/A

ACTION: If any holding times have been exceeded, qualify all results for the associated samples as estimated (J for detects and UJ for non-detects).

3. INSTRUMENT CALIBRATION AND TUNING

3.1 GC/MS TUNING

Is a DFTPP tune report present for each applicable 12h period?	Yes	No	N/A
Do all tunes on all instruments meet the tuning criteria?	Yes	No	N/A
Do all tunes on all instruments meet the expanded criteria?	Yes	No	N/A
Has the laboratory made any calculation or transciption errors?	Yes	No	N/A
Have the proper significant figures been reported?	Yes	No	N/A

ACTION: If the mass calibration is out of specification but within the expanded criteria, qualify associated data as estimated (J for detects and UJ for non-detects). If all tuning criteria are not met, qualify all associated data as unusable (R).

3.2 INITIAL CALIBRATION

Is an initial calibration report provided for all instruments?	Yes	No	N/A
Are all RSD values ≤30%?	Yes	No	N/A
Are all RRF values ≥0.05?	Yes	No	N/A

ACTION: If any RRF value is out of specification qualify all detected results for the particular compound as estimated (J) and all non-detects as unusable (R). If any RSD value is out of specification qualify all associated data as estimated (J for detects and UJ for non-detects).

3.3. CONTINUING CALIBRATION

Is a continuing calibration report present for all 12h periods in which associated samples were analyzed ?	Yes	No	N/A
Are all RRF values ≥0.05?	Yes	No	N/A
Are all %D values ≤25%?	Yes	No	N/A

ACTION: If any RRF value is out of specification qualify all associated detected results as estimated (J) and all non-detects as unusable (R). If any %D is out of specification, qualify all associated results as estimated (J for detects and UJ for non-detects).

4. BLANKS

4.1 LABORATORY BLANKS

Has the laboratory conducted a method blank analysis per matrix for every extraction batch?	Yes	No	N/A
Are compounds reported in the laboratory blanks?	Yes	No	N/A

ACTION: Qualify all sample results <10X the highest blank concentration for the common laboratory contaminants, as non-detects (U) or at the SQL if the result is <CRQL. Qualify all remaining sample results <5X the blank concentration in similar fashion.

4.2. FIELD BLANKS

Are compounds reported in the field blanks?	Yes	No	N/A

ACTION: Qualify all detected sample results less than or equal to five times the amount in any valid field blank as non-detects (U) and note the results of the field blanks in the validation narrative.

5. ACCURACY

5.1 SURROGATE RECOVERY

Are any surrogate recoveries out of specification?	Yes	No	N/A
Are any surrogate recoveries less than 10%?	Yes	No	N/A
Are any method blank surrogate recoveries out of specification?	Yes	No	N/A

ACTION: Qualify all associated data as estimated (J for detects and UJ for non-detects) if at least two semivolatile surrogates are out of specification. If any surrogate is below 10% recovery qualify associated detected results as estimated (J) and associated non-detect results as unusable (R). If method blank surrogates are out of specification and associated sample surrogates are acceptable no qualification is required, however, the laboratory should be contacted for an explanation.

5.2 MATRIX SPIKE RECOVERY

Has an MS/MSD analysis been conducted per matrix in the sample group?	Yes	No	N/A
Are MS/MSD recoveries within specification?	Yes	No	N/A
Are there any calculation errors?	Yes	No	N/A

ACTION: If MS/MSD analyses have not been conducted contact the laboratory for explanation. Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the results in the validation narrative. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

5.3 PERFORMANCE AUDIT SAMPLES

Are the results for the performance audit samples within the acceptance limits?	Yes	No	N/A

ACTION: Note the results of the performance audit samples in the validation narrative.

6. PRECISION

6.1 MATRIX SPIKE/MATRIX SPIKE DUPLICATES

Are all RPD values within specification? Yes No N/A

ACTION: Review the MS/MSD results in conjunction with other QC data such as field duplicates and note the results in the validation narrative. If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

6.2 FIELD DUPLICATE SAMPLES

Are field duplicate RPD values acceptable? Yes No N/A

ACTION: Note the results of the field duplicate samples in the validation narrative.

6.3 FIELD SPLIT SAMPLES

Are field split RPD values acceptable? Yes No N/A

ACTION: Note the results of the field split samples in the validation narrative.

7. SYSTEM PERFORMANCE

7.1 INTERNAL STANDARDS PERFORMANCE

Are any internal standard area counts outside the acceptance limits? Yes No N/A

Are retention times for any internal standard outside the ±30 second windows established by the most recent calibration check? Yes No N/A

ACTION: If the area counts are outside the acceptance limits qualify all associated results as estimated (J for detects and UJ for non-detects). If area counts are outside the acceptance limits and the retention time criteria are not met qualify all non-detects in the associated samples as unusable (R).

8. COMPOUND IDENTIFICATION AND QUANTITATION

8.1 COMPOUND IDENTIFICATION

Are detected compounds within ±0.06 relative retention time units of the associated calibration standard?	Yes	No	N/A
Are all ions at a relative intensity of ≥10% in the standard spectra present in the sample spectra?	Yes	No	N/A
Do the relative intensities between the standard and sample spectra agree within 20%?	Yes	No	N/A
Have all ions >10% in the sample spectra that are not present in the standard spectra been explained?	Yes	No	N/A

ACTION: If compound identification is in error and retention time and mass spectral criteria are exceeded qualify all affected positive results as unusable (R). If cross-contamination between analyses is suspected, qualify affected data as unusable (R).

8.2 REPORTED RESULTS AND QUANTITATION LIMITS

Has the laboratory used the correct RRF values and internal standards for quantitation?	Yes	No	N/A
Are results and quantitation limits calculated properly?	Yes	No	N/A
Has the laboratory reported the sample quantitation limits within five times the CRQL values?	Yes	No	N/A

ACTION: If the quantitation limits are in error contact the laboratory for clarification and note in the validation narrative.

8.3 TENTATIVELY IDENTIFIED COMPOUNDS

Has the laboratory conducted a spectral library search on all candidate TIC peaks in accordance with the analytical SOW?	Yes	No	N/A
Has the laboratory properly identified and coded all TIC?	Yes	No	N/A

ACTION: If the laboratory has failed to search the minimum number of TIC peaks in the chromatogram contact the laboratory for submittal of the required data. Qualify as non-detects (U) all TIC compounds present in samples and blanks using the review criteria specified in the validation requirements. If TIC identification is in error sample results should be qualified as non-detects (U) or unusable (R). If TIC identifications are judged valid, qualify the results as presumptive and estimated (JN).

9. OVERALL ASSESSMENT AND SUMMARY

Has the laboratory conducted the analysis in accordance with the analytical SOW?	Yes	No	N/A
Were project specific data quality objectives met for this analysis?	Yes	No	N/A

ACTION: Summarize all the data qualifications and complete the data validation narrative as specified in Section 10 of the data validation requirements.

COMMENTS (attach additional sheets as necessary):

PESTICIDE/PCB DATA REVIEW CHECKLIST - FORM A-3

PROJECT:	REVIEWER:	DATE:
LABORATORY:	CASE:	SDG:
SAMPLES/MATRIX:		

1. DATA PACKAGE COMPLETENESS

Review the data package for completeness and check off the items below. If any data review elements are missing contact the laboratory for re-submittal.

Data Package Item	Present?:	Yes	No	N/A
Case Narrative		—	—	—
Data Summary		—	—	—
Chain-of-Custody		—	—	—
QC Summary				
Surrogate report		—	—	—
MS/MSD report		—	—	—
Blank summary report		—	—	—
Sample Data				
Sample reports		—	—	—
Chromatograms		—	—	—
GC integration reports		—	—	—
Worksheets		—	—	—
UV traces from GPC		—	—	—
GC/MS confirmation spectra		—	—	—
Standards Data				
Pesticides Evaluation Standards Summary		—	—	—
Pesticides/PCB Standards Summary		—	—	—
Pesticides/PCB identification		—	—	—
Pesticides standard chromatograms		—	—	—
Raw QC Data				
Blank analysis report forms and chromatograms		—	—	—
MS/MSD report forms and chromatograms		—	—	—

Data Package Item	Present?:	Yes	No	N/A
Additional Data				
Moisture/% solids data sheets		___	___	___
Reduction formulae		___	___	___
Instrument time logs		___	___	___
Chemist notebook pages		___	___	___
Sample preparation sheets		___	___	___

2. HOLDING TIMES

Were all samples extracted within holding time?	Yes	No	N/A
Were all samples analyzed within holding time?	Yes	No	N/A

Action: If any holding times were exceeded, qualify all affected sample results as estimated (J for detects and UJ for non-detects).

3. INSTRUMENT PERFORMANCE AND CALIBRATIONS

3.1 INSTRUMENT PERFORMANCE

Are DDT retention times greater than 12 minutes?	Yes	No	N/A
Is resolution between DDT peaks acceptable?	Yes	No	N/A
Do all pesticide standards elute within the established retention time windows?	Yes	No	N/A
Are DDT breakdowns <20%?	Yes	No	N/A
Are endrin breakdowns <20%?	Yes	No	N/A
Are DBC retention time differences within specification?	Yes	No	N/A

ACTION: If DDT retention time is <12 minutes and resolution is ≤25% qualify associated data as unusable (R). If the standards do not meet the retention time criteria and peaks are not present near or within the retention time windows no sample qualification is necessary. If peaks are near or within the retention time windows and the standards and matrix spikes do not fall within the expanded retention time windows calculated according to the validation requirements, qualify all associated sample results from the last in-control point as unusable (R). If the DDT percent breakdown exceeds 20%, qualify all detected results for DDT as estimated (J) and all non-detects as unusable (R) if DDD and DDE are detected. In addition qualify all results for DDD or DDE as presumptive and estimated (NJ). If the endrin breakdown exceeds 20%, qualify all detected results for endrin as estimated (J) and all non-

detects as unusable (R) if endrin aldehyde or endrin ketone are detected. In addition qualify all results for endrin ketone as presumptive and estimated (NJ). If DBC %D values are outside the limits and the shift is ocurring repeatedly in samples and standards, qualify affected sample results as unusable (R).

3.2 CALIBRATION

Are RSD values for aldrin, endrin, DDT and DBC ≤10%?	Yes	No	N/A
Have all standards been analyzed within 72 hours of any sample?	Yes	No	N/A
Has a 3-point calibration been conducted for DDT or toxaphene?	Yes	No	N/A
Have all standards been analyzed at the start of each 72h sequence?	Yes	No	N/A
Have evaluation standards A, B, and C been analyzed within 72h of any sample?	Yes	No	N/A
Has the confirmation standard mix been analyzed after every 5 samples?	Yes	No	N/A
Has evaluation standard B analyzed every 10 samples?	Yes	No	N/A
Are %D values for initial and subsequent standards ≤15% for quantitation standards and ≤20% for confirmation standards?	Yes	No	N/A

ACTION: If the RSD criteria were exceeded or three point calibrations not conducted qualify associated detects as estimated (J). If all standards were not analyzed at the beginning of each 72h sequence qualify associated data as unusable (R). If the confirmation standards were not analyzed properly qualify associated detects as estimated (J). If the continuing calibration criteria were not met qualify associated quantitation data as estimated (J).

4. BLANKS

4.1 LABORATORY BLANKS

Has the laboratory analyzed the method blanks for each matrix in the sample group?	Yes	No	N/A
Are target compounds present in the laboratory blanks?	Yes	No	N/A

ACTION: Qualify all associated positive results as non-detects (U) that are <5X the highest concentration in any acceptable blank.

4.2 FIELD BLANKS

Are target compounds present in the field blanks? Yes No N/A

ACTION: If target compounds are present in the field blanks qualify all positive sample results <5X the highest valid field blank concentrations as non-detects (U) and note the results in the validation narrative.

5. ACCURACY

5.1 SURROGATE RECOVERY

Are any surrogate recoveries out of specification? Yes No N/A

Do any samples show non-detects for surrogates? Yes No N/A

Are any method blank surrogates out of specification? Yes No N/A

ACTION: Qualify all associated sample results as estimated (J for detects and UJ for non-detects) for surrogates out of specification. If the surrogate was not detected (0% recovery) in the sample qualify associated non-detects as unusable (R). If method blank surrogates are out of specification and sample surrogates are acceptable, no qualification is required however, the laboratory should be contacted for an explanation.

5.2 MATRIX SPIKE RECOVERY

Has the laboratory analyzed a MS/MSD per matrix for the the sample group? Yes No N/A

Are MS/MSD recoveries within specification? Yes No N/A

Are there any calculation or transcription errors? Yes No N/A

ACTION: If MS/MSD analyses have not been conducted contact the laboratory for clarification. Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the results in the validation narrative. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

5.3 PERFORMANCE AUDIT SAMPLES

Are performance audit sample results within the acceptance limits? Yes No N/A

ACTION: Note the results of the performance audit samples in the validation narrative.

6. PRECISION

6.1 MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES

Are the RPD values within specification? Yes No N/A

ACTION: Review the MS/MSD results in conjunction with other QC data such as field duplicates and not the results in the validation narrative. If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

6.2 FIELD DUPLICATE SAMPLES

Are field duplicate RPD values acceptable? Yes No N/A

ACTION: Note the results of the field duplicate samples in the validation narrative.

6.3 FIELD SPLIT SAMPLES

Are field split RPD values acceptable? Yes No N/A

ACTION: Note the results of the field split samples in the validation narrative.

7. COMPOUND IDENTIFICATION AND QUANTITATION

7.1 COMPOUND IDENTIFICATION

Do positive results meet the retention time window criteria? Yes No N/A

Were positive results analyzed on disimilar columns? Yes No N/A

If dieldrin and DDE were reported was a 3% OV-1 column used for confirmation? Yes No N/A

Do retention times and relative peak height ratios match the expected patterns for multipeak compounds (PCB, toxaphene or chlordane)? Yes No N/A

Has GC/MS confirmation been conducted on sample extract concentrations >10 ppm? Yes No N/A

ACTION: If positive results do not meet the retention time criteria qualify all detected results as non-detects as follows: If the misidentified peak is outside the retention time windows and no interferences are noted report the CRQL and if the misidentified peak interferes with a target peak then the report value is qualified as estimated and non-detected (UJ). If

positive results were not confirmed on disimilar columns, reject affected results (R). If a 3% OV-1 was used to confirm dieldrin and DDE, reject the affected data (R). If PCB, chlordane or toxaphene identification is questionable qualify the results as presumptive and estimated (NJ). If GC/MS confirmation was not conducted contact the laboratory for explanation and note in the validation narrative.

7.2 REPORTED RESULTS AND QUANTITATION LIMITS

Are results and quantitation limits calculated properly?	Yes	No	N/A
Has the laboratory reported the sample quantitation limits within five times the CRQL values?	Yes	No	N/A

ACTION: If results and quantitation limits are in error contact the laboratory for clarification and note in the validation narrative.

8. OVERALL ASSESSMENT AND SUMMARY

Has the laboratory conducted the analysis in accordance with the analytical SOW?	Yes	No	N/A
Were project specific data quality objectives met for this analysis?	Yes	No	N/A

ACTION: Summarize all the data qualifications and complete the data validation narrative as specified in Section 10 of the data validation requirements.

COMMENTS (attach additional sheets as necessary):

HERBICIDE DATA REVIEW CHECKLIST - FORM A-4

PROJECT:	REVIEWER:	DATE:
LABORATORY:	CASE:	SDG:
SAMPLES/MATRIX:		

1. DATA PACKAGE COMPLETENESS

Review the data package for completeness and check off the items below. If any data review elements are missing contact the laboratory for submittal.

Data Package Item	Present?:	Yes	No	N/A
Case Narrative		___	___	___
Data Summary		___	___	___
Chain of Custody Forms		___	___	___
Sample Analysis Request		___	___	___
QC Summary				
Surrogate Recovery		___	___	___
MS/MSD Recovery		___	___	___
Method Blank Summary		___	___	___
Sample Data				
Sample Results		___	___	___
Chromatograms for all samples/extracts		___	___	___
Quantitation sheets for all samples/extracts		___	___	___
Extraction data sheets for all samples/extracts		___	___	___
Instrument time/run logs for all samples/extracts		___	___	___
Standards Data				
Initial Calibration standard concentrations		___	___	___
Initial Calibration summary of RRF/RSD data		___	___	___
Chromatograms for all initial cal. standards		___	___	___
Quantitation sheets for all initial cal. standards		___	___	___
Instrument time/run logs for all samples/extracts		___	___	___
Calibration standard traceability data		___	___	___

Data Package Item	Present?:	Yes	No	N/A
Raw QC Data				
Blanks				
Laboratory Blank results		___	___	___
Chromatograms for all laboratory blanks		___	___	___
Quantitation reports for all laboratory blanks		___	___	___
Matrix Spike/Matrix Spike Duplicates				
MS/MSD Results		___	___	___
Chromatograms		___	___	___
Quantitation reports		___	___	___
Additional Data				
Moisture/% Solids data sheets		___	___	___
Calculation formulae		___	___	___
Instrument Run/Time Logs		___	___	___
Chemist notebook pages		___	___	___
Sample preparation sheets		___	___	___

2. HOLDING TIMES

Were all samples extracted within holding times?	Yes	No	N/A
Were all samples analyzed within holding times?	Yes	No	N/A

ACTION: If the extraction or analytical holding times were exceeded, qualify all affected results as estimated (J for detects and UJ for non-detects).

3. INSTRUMENT CALIBRATION

3.1 INITIAL CALIBRATION

Was an initial calibration conducted prior to sample analysis?	Yes	No	N/A
Are all RSD values less than 20%?	Yes	No	N/A

ACTION: If the RSD criteria were not met, qualify all results as estimated (J for detects and UJ for non-detects).

3.2 CONTINUING CALIBRATION

Have continuing calibrations been conducted at the proper frequency?	Yes	No	N/A
Are the RRFs within ±15% of the initial calibration average RF?	Yes	No	N/A
Are the RT values for the calibration compounds within the retention time windows?	Yes	No	N/A

ACTION: If the percent difference criteria or retention time windows are not met, qualify all associated data as estimated (J for detects, UJ for non-detects).

4. BLANKS

4.1 LABORATORY BLANKS

Has the laboratory analyzed at least one method blank per matrix in the sample batch?	Yes	No	N/A
Are target compounds present in the laboratory blanks?	Yes	No	N/A

ACTION: Qualify all detected results in the samples that are <5X the amount in any laboratory blank as non-detects (U).

4.2 FIELD BLANKS

Are target compounds present in the field blanks?	Yes	No	N/A

ACTION: Qualify all detected results in the samples that are <5X the amount in any valid field blank as non-detects (U).

5. ACCURACY

5.1 SURROGATE RECOVERY

Are any surrogate recoveries out of specification?	Yes	No	N/A
Are any surrogates non-detected?	Yes	No	N/A

ACTION: Surrogate recoveries out of specification will require qualification of all associated data as estimated (J for detects and UJ for non-detects). Surrogate recoveries that are 0% will require qualification of all detects as estimated (J) and the rejection of all non-detects (R).

5.2 MATRIX SPIKE RECOVERY

Has the laboratory conducted a MS/MSD analysis per matrix for the sample group?	Yes	No	N/A
Are there calculation or transcription errors?	Yes	No	N/A
Are MS recoveries within specification?	Yes	No	N/A

ACTION: If MS/MSD analyses have not been conducted contact the laboratory for clarification. Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the results in the validation narrative. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

5.3 PERFORMANCE AUDIT SAMPLES

Are performance audit sample results within the acceptance limits?	Yes	No	N/A

ACTION: Note the results of the performance audit samples in the validation narrative.

6. PRECISION

6.1 MATRIX SPIKE/MATRIX SPIKE DUPLICATES

Are there any calculation or transcription errors?	Yes	No	N/A
Are the RPD values within specification?	Yes	No	N/A

ACTION: Review the MS/MSD results in conjunction with other QC data such as field duplicates and not the results in the validation narrative. If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

6.2 FIELD DUPLICATES

Are the field duplicate RPDs acceptable?	Yes	No	N/A

ACTION: Note the results of the field duplicate samples in the validation narrative.

6.3 FIELD SPLIT SAMPLES

Are the field split RPDs acceptable? Yes No N/A

ACTION: Note the results of the field split samples in the validation narrative.

7. COMPOUND IDENTIFICATION AND QUANTITATION

7.1 COMPOUND IDENTIFICATION

Are positive results within the retention time windows? Yes No N/A

Are positive results unaffected by interfering peaks? Yes No N/A

ACTION: If positive results are not within the retention time windows qualify all detected results as non-detects as follows: If the misidentified peak is outside the retention time windows and no potential interferences are present, report the CRQL and if the misidentified peak interferes with the potential detection of a target peak then the reported value is the quantitation limit and the result is qualified as estimated (UJ).

7.2 REPORTED RESULTS AND QUANTITATION LIMITS

Has the laboratory reported sample quantitation limits within five times the CRQL levels? Yes No N/A

Are there any calculation or transcription errors? Yes No N/A

ACTION: If the results and quantitation limits are in error contact the laboratory for clarification and discuss in the validation narrative.

8. OVERALL ASSESSMENT AND SUMMARY

Has the laboratory conducted the analysis in accordance with the analytical SOW? Yes No N/A

Were project specific data quality objectives met for this analysis? Yes No N/A

ACTION: Summarize all the data qualifications and complete the data validation narrative as specified in Section 10 of the data validation requirements.

COMMENTS (attach additional sheets as necessary):

DIOXIN/FURAN DATA REVIEW CHECKLIST - FORM A-5

PROJECT:	REVIEWER:	DATE:
LABORATORY:	CASE:	SDG:
SAMPLES/MATRIX:		

1. DATA PACKAGE COMPLETENESS

Review the data package for completeness and check off the items below. If any data review elements are missing contact the laboratory for submittal.

Data Package Item	Present?:	Yes	No	N/A
Case Narrative		___	___	___
Data Summary		___	___	___
Chain of Custody Forms		___	___	___
Sample Analysis Request Forms		___	___	___
QC Summary				
MS tuning information		___	___	___
Internal standards recovery		___	___	___
MS/MSD Recovery		___	___	___
Method Blank Summary		___	___	___
Sample Data				
Sample Results		___	___	___
Chromatograms for all samples/extracts		___	___	___
Quantitation sheets for all samples/extracts		___	___	___
Extraction data sheets for all samples/extracts		___	___	___
Instrument time/run logs all samples/extracts		___	___	___
Standards Data				
Calibration standard concentrations		___	___	___
Initial Calibration summary of RRF/RSD data		___	___	___
Initial Calibration summary of isotope ratios		___	___	___
Chromatograms for all initial cal. standards		___	___	___
Quantitation sheets for all initial cal. standards		___	___	___
Continuing calibration summary of RRF/%D data		___	___	___
Continuing calibration summary of isotope ratios		___	___	___
Chromatograms for all cont. cal. standards		___	___	___

Data Package Item	Present?:	Yes	No	N/A
Quantitation sheets for all cont. cal. standards		—	—	—
Instrument time/run logs for all standards		—	—	—
Calibration standard traceability data		—	—	—
Raw QC Data				
Blanks				
Laboratory Blank results		—	—	—
Chromatograms for all laboratory blanks		—	—	—
Quantitation reports for all laboratory blanks		—	—	—
Matrix Spike/Matrix Spike Duplicates				
MS/MSD Results		—	—	—
Chromatograms		—	—	—
Quantitation reports		—	—	—
Additional Data				
Moisture/% Solids data sheets		—	—	—
Calculation formulae		—	—	—
Chemist notebook pages		—	—	—
Sample preparation sheets		—	—	—

2. HOLDING TIMES

Were all samples extracted within holding times?	Yes	No	N/A
Were all samples analyzed within holding times?	Yes	No	N/A

ACTION: If the holding times were exceeded, qualify all affected results as estimated (J for detects and UJ for non-detects).

3. INSTRUMENT PERFORMANCE AND CALIBRATION

3.1 GC COLUMN PERFORMANCE

Is chromatographic resolution for TCDD acceptable?	Yes	No	N/A
Has the laboratory analyzed a performance check solution at the required frequency?	Yes	No	N/A
Has the laboratory established proper SIM conditions for each isomer series?	Yes	No	N/A

ACTION: If the column performance criteria are not met qualify associated data as unusable (R).

3.2 MS PERFORMANCE

Did the laboratory tune the MS prior to sample analysis?	Yes	No	N/A
Was MS resolution acceptable (≥10,000) prior to sample analysis?	Yes	No	N/A

ACTION: If the laboratory failed to achieve the resolution requirements prior to sample analysis reject all associated data (R).

3.3 INITIAL CALIBRATION

Was an acceptable initial calibration conducted prior to sample analysis?	Yes	No	N/A
Are all RSDs ≤20% for unlabeled standards?	Yes	No	N/A
Are all RSDs ≤30% for labeled standards?	Yes	No	N/A
Are chlorine isotope ratios within specification?	Yes	No	N/A
Are signal to noise ratios in all calibrations ≥2.5?	Yes	No	N/A
Are there any calculation or transcription errors?	Yes	No	N/A

ACTION: If any criteria were exceeded, qualify all associated results as estimated (J for detects and UJ for non-detects).

3.4 CONTINUING CALIBRATION

Have acceptable continuing calibrations been conducted at the proper frequency?	Yes	No	N/A
Are the RSD values ≤20% for unlabeled standards?	Yes	No	N/A
Are the RSD values for labeled standards ≤30%?	Yes	No	N/A

ACTION: If any criteria were exceeded qualify associated results as estimated (J for detects and UJ for non-detects).

4. BLANKS

4.1 LABORATORY BLANKS

Has the laboratory analyzed at least one method blank per matrix in the sample batch?	Yes	No	N/A
Are target compounds present in the laboratory blanks?	Yes	No	N/A

ACTION: If no method blank was analyzed and reported for the sample delivery group, contact the laboratory for submittal of the required information. Qualify all detected results in the samples that are <5X the amount in any laboratory blank as non-detects (U).

4.2 FIELD BLANKS

Are target compounds present in the field blanks?	Yes	No	N/A

ACTION: Qualify all detected results in the samples that are <5X the amount in any valid field blank as non-detects (U).

5. ACCURACY

5.1 MATRIX SPIKE RECOVERY

Has the laboratory conducted a matrix spike/matrix spike duplicate analysis per matrix for the sample group?	Yes	No	N/A
Are there any transcription or calculation errors?	Yes	No	N/A
Are MS recoveries within 40 to 140%?	Yes	No	N/A

ACTION: If the laboratory has not conducted the requisite MS/MSD analyses, review the sample request forms and chain-of-custody for discrepancies and contact the laboratory for clarification. Review the MS/MSD recoveries in conjunction with other QC data such as surrogate recoveries and note the results in the validation narrative. If it is determined from the review that out of specification MS/MSD recoveries are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

5.2 PERFORMANCE AUDIT SAMPLES

Are performance audit sample results within the acceptance limits?	Yes	No	N/A

ACTION: Note the results of the performance audit samples in the validation narrative.

6. PRECISION

6.1 MATRIX SPIKE/MATRIX SPIKE DUPLICATES

Are the RPD values within specification? Yes No N/A

ACTION: Review the MS/MSD results in conjunction with other QC data such as field duplicates and not the results in the validation narrative. If it is determined from the review that out of specification MS/MSD results are indicative of systematic problems in the laboratory such as sample preparation or sample-specific matrix interferences this must be noted in the validation narrative along with the potential affect on the sample results.

6.2 FIELD DUPLICATES

Are the field duplicate RPDs acceptable? Yes No N/A

ACTION: Note the results of the field duplicate samples in the validation narrative.

6.3 FIELD SPLIT SAMPLES

Are the field split RPDs acceptable? Yes No N/A

ACTION: Note the results of the field split samples in the validation narrative.

7. SYSTEM PERFORMANCE

7.1 INTERNAL STANDARDS PERFORMANCE

Are internal standard recoveries within 40 to 120%? Yes No N/A

ACTION: If internal standard recoveries are out of specification, qualify associated positive results as estimated (J). If internal standard peak resolution is low (≤25%), qualify associated non-detects as unusable (R), otherwise qualify associated non-detects as estimated (UJ).

8. COMPOUND IDENTIFICATION AND QUANTITATION

8.1 COMPOUND IDENTIFICATION

Has the laboratory monitored the specified masses during analysis?	Yes	No	N/A
Are PCDPE interferences present?	Yes	No	N/A
Do positive sample results show ≥2.5 signal to noise?	Yes	No	N/A
Are chlorine isotope ratios acceptable?	Yes	No	N/A
Are positive results within the RT windows of the associated internal standard?	Yes	No	N/A

ACTION: If the laboratory has not monitored the required ions, reject data for associated congeners (R). If PCDPE interferences are present at ≥2.5 signal to noise ratio, qualify associated congeners as estimated (J for detects and UJ for non-detects). If positive results do not meet the signal to noise requirements, qualify all associated data as estimated (J for detects and UJ for non-detects). If the chlorine isotope ratios are exceeded for positive results, discuss in the validation narrative. If positive results do not meet the RT windows of the associated internal standard qualify all associated data as unusable (R).

8.2 REPORTED RESULTS AND QUANTITATION LIMITS

Has the laboratory reported sample quantitation limits within five times the work plan CRQL levels?	Yes	No	N/A
Are there any calculation or transcription errors?	Yes	No	N/A

ACTION: If the laboratory was unable to meet CRQLs within a factor of five and no explanation has been provided in the case narrative, contact the laboratory for clarification and note in the validation narrative.

9. OVERALL ASSESSMENT AND SUMMARY

Has the laboratory conducted the analysis in accordance with the analytical SOW?	Yes	No	N/A
Were project specific data quality objectives met for this analysis?	Yes	No	N/A

ACTION: Summarize all the data qualifications and complete the data validation narrative as specified in Section 10 of the data validation requirements.

COMMENTS (attach additional sheets as necessary):

INORGANIC ANALYSIS DATA REVIEW CHECKLIST - FORM A-6

PROJECT:	REVIEWER:	DATE:
LABORATORY:	CASE:	SDG:
SAMPLES/MATRIX:		

1. COMPLETENESS AND CONTRACT COMPLIANCE

Review the data package for completeness and check off the items below. If any data review elements are missing contact the laboratory for submittal of the omitted data.

Data Package Item	Present?:	Yes	No	N/A
Case Narrative		___	___	___
Cover Page		___	___	___
Traffic Reports		___	___	___
Sample Data				
Inorganic Analysis Data Sheets		___	___	___
Standards Data				
Initial and Continuing Calibration Verification		___	___	___
CRDL Standard for AA and ICP		___	___	___
QC Summary				
Blanks		___	___	___
ICP Interference Check Summary		___	___	___
Spike Sample Recovery		___	___	___
Post-Digestion Spike Sample Recovery		___	___	___
Duplicate		___	___	___
Laboratory Control Sample		___	___	___
Standard Addition Results		___	___	___
ICP Serial Dilutions		___	___	___
Instrument Detection Limits		___	___	___
ICP Interelement Correction Factors		___	___	___
ICP Linear Ranges		___	___	___
Preparation Log		___	___	___
Analysis Run Log		___	___	___

Data Package Item	Present?:	Yes	No	N/A
Raw Data				
ICP Raw Data		___	___	___
Furnace AA Raw Data		___	___	___
Mercury Raw Data		___	___	___
Cyanide Raw Data		___	___	___
Additional Data				
Internal laboratory chain-of-custody		___	___	___
Laboratory Sample Preparation Records		___	___	___
Percent Solids Analysis Records		___	___	___
Reduction Formulae		___	___	___
Instrument Run Logs		___	___	___
Chemist Notebook Pages		___	___	___

2. HOLDING TIMES

Have all samples been analyzed within holding times? Yes No N/A

ACTION: If any holding times have been exceeded qualify all affected results as estimated (J for detects and UJ for non-detects).

3. INITIAL CALIBRATIONS

Were all instruments calibrated daily, each set-up time and were the proper number of standards used? Yes No N/A

Are the correlation coefficients ≥0.995? Yes No N/A

Was a midrange CN standard distilled? Yes No N/A

ACTION: Qualify all data as unusable if reported from an analysis in which an instrument was not calibrated or was calibrated with less than the minimum number of standards. Qualify associated sample results > IDL as estimated (J) and results < IDL as estimated (UJ), if the correlation coefficient is < 0.995 or the laboratory did not distill the midrange CN standard.

4. INITIAL AND CONTINUING CALIBRATION VERIFICATION

Are ICV and CCV percent recoveries within control? Yes No N/A

Are there calculation errors? Yes No N/A

ACTION: Qualify all affected data in accordance with Section 8.3 of the validation requirements. If calculation errors are noted, contact the laboratory for clarification.

5. ICP INTERFERENCE CHECK SAMPLE

Has an ICS sample been analyzed at the proper frequency?	Yes	No	N/A
Are the AB solution %R values within control?	Yes	No	N/A
Are there calculation errors?	Yes	No	N/A

ACTION: Qualify all affected data in accordance with Section 8.3 of the validation requirements. If calculation errors are noted, contact the laboratory for clarification.

6. LABORATORY BLANKS

Are target analytes present in the field blanks?	Yes	No	N/A

ACTION: Qualify all associated sample results for any analyte <5X the amount in any laboratory blank as non-detected (U).

7. FIELD BLANKS

Are target analytes present in the field blanks?	Yes	No	N/A

ACTION: Qualify all sample results for any analyte <5X the amount in any valid field blank as non-detected (U).

8. MATRIX SPIKE SAMPLE ANALYSIS

Are spike recoveries within the control limits?	Yes	No	N/A

ACTION: Qualify the affected sample data according to the following requirements:

If spike recovery is >125% and sample results are <IDL no qualification is required. If spike recovery is >125% or <75% qualify all positive results as estimated (J). If spike recovery is 30% to 74% qualify all non-detects as estimated (UJ). If spike recovery is <30%, reject all non-detects (R).

9. LABORATORY CONTROL SAMPLE

Are percent recoveries within the acceptance limits?	Yes	No	N/A
Are there calculation errors?	Yes	No	N/A

ACTION: Qualify the sample data according to the following requirements:

AQUEOUS LCS - Qualify as estimated (J), all sample results > IDL, for which the LCS %R falls within the range 50-79% or > 120%. Qualify as estimated (UJ), all sample results < IDL, for which the LCS falls within the range of 50-79%. Qualify as unusable (R) all sample results, for which the LCS %R <50%.

SOLID LCS - Qualify as estimated (J), all sample results > IDL for which the LCS result is outside the established control limits. Qualify as estimated (UJ), all sample results < IDL for which the LCS %R are lower than the established control limits.

10. PERFORMANCE AUDIT ANALYSES

Are the performance audit sample results within the acceptance limits?	Yes	No	N/A

ACTION: Note the results of the performance audit sample analyses in the data validation narrative.

11. DUPLICATE SAMPLE ANALYSIS

Are RPD values acceptable?	Yes	No	N/A

ACTION: Qualify the results for all associated samples of the same matrix as estimated (J) if the RPD results fall outside the appropriate control limits.

12. ICP SERIAL DILUTION

Are the serial dilution results acceptable?	Yes	No	N/A
Is there evidence of negative interference?	Yes	No	N/A

ACTION: Qualify the associated data as estimated (J) for those analytes in which the %D is outside the control limits. If evidence of negative interference is found, use professional judgment to qualify the data.

13. FIELD DUPLICATE SAMPLES

Do the RPD values exceed the control limits?	Yes	No	N/A

ACTION: Note the results of the field duplicate samples in the validation narrative.

14. FIELD SPLIT SAMPLES

Do the RPD values exceed the control limits?	Yes	No	N/A

ACTION: Note the results of the field split samples in the validation narrative.

15. FURNACE ATOMIC ABSORPTION QUALITY CONTROL

Do all applicable analyses have duplicate injections?	Yes	No	N/A
Are applicable duplicate injection RSD values within control?	Yes	No	N/A
If no, were samples rerun once as required?	Yes	No	N/A
Does the RSD for the rerun fall within the control limits?	Yes	No	N/A
Were analytical spike recoveries within the control limits?	Yes	No	N/A
If no, were MSA analyses performed when required?	Yes	No	N/A
Are MSA correlation coefficients $\geq$0.995?	Yes	No	N/A
If no, was a second MSA analysis performed?	Yes	No	N/A

ACTION: If duplicate injections are outside the acceptance limits and the sample has not been reanalyzed or the reanalysis is outside the acceptance limits, qualify the associated data as estimated (J for detects and UJ for non-detects). If the analytical spike recovery is less than 40 percent qualify detects as estimated (J). If the analytical spike recovery is greater than or equal to 10% but less than 40 percent, qualify all non-detects as estimated (UJ) and if the analytical spike recovery is less than 10 percent, reject all non-detects (R). If the sample absorbance is less than 50% of the analytical spike absorbance and the analytical spike recovery is less than 85% or greater than 115%, qualify all results as estimated (J for detects and UJ for non-detects). If method of standard additions (MSA) was required but was not performed, the MSA samples were spiked incorrectly, or the MSA correllation coefficient was less than 0.995, qualify the associated detected results as estimated (J).

16. ANALYTE QUANTITATION AND DETECTION LIMITS

Have results been reported and calculated correctly?	Yes	No	N/A
Are results within the calibrated range of the instruments and within the linear range of the ICP?	Yes	No	N/A
Are all detection limits below the CRQL?	Yes	No	N/A

Action: If analyte quantitation is in error, contact the laboratory for explanation. If errors or deficiencies can not be resolved with the laboratory, qualify associated data as unusable (R).

17. OVERALL ASSESSMENT AND SUMMARY

Has the laboratory conducted the analysis in accordance with the analytical SOW?	Yes	No	N/A
Were project specific data quality objectives met for this analysis?	Yes	No	N/A

ACTION: Summarize all the data qualifications and complete the data validation narrative as specified in Section 10 of the data validation requirements.

COMMENTS (attach additional sheets as necessary):

WET CHEMISTRY DATA REVIEW CHECKLIST - FORM A-7

PROJECT:	REVIEWER:	DATE:
LABORATORY:	CASE:	SDG:
SAMPLES/MATRIX:		

1. DATA PACKAGE COMPLETENESS

Review the data package for completeness and check off the items below. If any data review elements are missing contact the laboratory for submittal of the omitted data.

Data Package Item	Present?:	Yes	No	N/A
Case Narrative		___	___	___
Cover Page		___	___	___
Traffic Reports/Chain-of-Custody		___	___	___
Sample Analysis Data Report Forms		___	___	___
Standards Data		___	___	___
QC Summary				
Blanks Summary Report Forms		___	___	___
Spike Sample Recovery Report Forms		___	___	___
Duplicate Sample Analysis Report Forms		___	___	___
Laboratory Control Sample Report Forms		___	___	___
Raw Data				
Ion Chromatograph Chromatograms		___	___	___
TOC and TOX Instrument Printouts		___	___	___
Laboratory Bench Sheets		___	___	___
Additional Data				
Laboratory Sample Preparation Logs		___	___	___
Instrument Run Logs		___	___	___
Internal Laboratory Chain-of-Custory		___	___	___
Percent Solids Analysis Records		___	___	___
Reduction Formulae		___	___	___
Chemist Notebook Pages		___	___	___

2. HOLDING TIMES

Were all samples analyzed within holding times? Yes No N/A

Action: If any holding times were exceeded qualify all affected results as estimated (J for detects and UJ for non-detects).

3. INITIAL CALIBRATIONS

Were all instruments calibrated daily, each set-up time and were the proper number of standards used? Yes No N/A

Are the correlation coefficients ≥0.995? Yes No N/A

Was a balance check conducted prior to the TDS analysis? Yes No N/A

Was the titrant normality checked? Yes No N/A

ACTION: Qualify all data as unusable (R) if reported from an analysis in which the above criteria were not met.

4. INITIAL AND CONTINUING CALIBRATION VERIFICATION

Are ICV and CCV percent recoveries within control? Yes No N/A

Are there calculation errors? Yes No N/A

ACTION: Qualify all affected data in accordance with the validation requirements.

5. LABORATORY BLANKS

Are target analytes present in the field blanks? Yes No N/A

ACTION: Qualify all associated sample results for any analyte <5X the amount in any laboratory blank as non-detected (U) and list the affected samples and analytes below.

6. FIELD BLANKS

Are target analytes present in the field blanks? Yes No N/A

ACTION: Qualify all sample results for any analyte <5X the amount in any valid field blank as non-detected (U).

7. MATRIX SPIKE SAMPLE ANALYSIS

Are spike recoveries within the acceptance limits? Yes No N/A

ACTION: If spike recovery is outside the control limits and the sample results are greater than the CRQL, qualify the data as estimated (J). If the spike recovery is less than 30% and the sample results are less then the IDL qualify the data as unusable (R).

8. LABORATORY CONTROL SAMPLE

Are percent recoveries within the acceptance limits? Yes No N/A

Are there calculation errors? Yes No N/A

ACTION: Qualify the affected results according to the following requirements:

AQUEOUS LCS - Qualify as estimated (J), all sample results > IDL, for which the LCS %R falls within the range 50-79% or > 120%. Qualify as estimated (UJ), all sample results < IDL, for which the LCS falls within the range of 50-79%. Qualify as unusable (R) all sample results, for which the LCS %R <50%.

SOLID LCS - Qualify as estimated (J), all sample results > IDL for which the LCS %R is outside the established control limits. Qualify as estimated (UJ), all sample results < IDL for which the LCS %R are lower than the established control limits.

9. PERFORMANCE AUDIT ANALYSES

Are the performance audit sample results within the acceptance limits? Yes No N/A

ACTION: Note the results of the performance audit samples in the validation narrative.

10. DUPLICATE SAMPLE ANALYSIS

Are RPD values within the acceptance limits? Yes No N/A

Action: Qualify the results for all associated samples of the same matrix as estimated (J) if the RPD falls outside the acceptance limits.

11. FIELD DUPLICATE SAMPLES

Do RPD values exceed the acceptance limits? Yes No N/A

ACTION: Note the results of the field duplicate samples in the validation narrative.

12. FIELD SPLIT SAMPLES

Do RPD values exceed the acceptance limits? Yes No N/A

ACTION: Note the results of the field split samples in the validation narrative.

13. ANALYTE QUANTITATION AND DETECTION LIMITS

Have results been reported and calculated correctly? Yes No N/A

Are instrument detection limits below the CRDL? Yes No N/A

Action: If analyte quantitation is in error, contact the laboratory for explanation. If errors or deficiencies can not be resolved with the laboratory, qualify associated data as unusable (R).

14. OVERALL ASSESSMENT AND SUMMARY

Has the laboratory conducted the analysis in accordance with the analytical SOW? Yes No N/A

Were project specific data quality objectives met for this analysis? Yes No N/A

ACTION: Summarize all the data qualifications and complete the data validation narrative as specified in Section 10 of the data validation requirements.

COMMENTS (attach additional sheets as necessary):

APPENDIX B

DATA SUMMARY FORMS

HOLDING TIME SUMMARY - FORM B-1

SDG:	REVIEWER:			DATE:		PAGE___OF___	
COMMENTS:							
FIELD SAMPLE ID	ANALYSIS TYPE	DATE SAMPLED	DATE PREPARED	DATE ANALYZED	PREP. HOLDING TIME, DAYS	ANALYSIS HOLDING TIME, DAYS	QUALIFIER

CALIBRATION DATA SUMMARY - FORM B-2

SDG:	REVIEWER:	DATE:		PAGE____OF____	
COMMENTS:					
CALIB. TYPE:	INITIAL CONTINUING	INSTRUMENT:			
CALIB. DATE	COMPOUND	RF	RSD/%D/%R	SAMPLES AFFECTED	QUALIFIER

BLANK AND SAMPLE DATA SUMMARY - FORM B-3

SDG:	REVIEWER:	DATE:				PAGE_____OF_____	
COMMENTS:							
SAMPLE ID	COMPOUND	RESULT	Q	RT	UNITS	SAMPLES AFFECTED	QUALIFIER

ACCURACY DATA SUMMARY - FORM B-4

SDG:	REVIEWER:	DATE:	PAGE___OF___	
COMMENTS:				
SAMPLE ID	COMPOUND	% RECOVERY	SAMPLE(S) AFFECTED	QUALIFIER REQUIRED

PRECISION DATA SUMMARY - FORM B-5

SDG:	REVIEWER	DATE:	PAGE___OF___		
COMMENTS:					
COMPOUND	SAMPLE ID:	SAMPLE ID:	RPD	SAMPLES AFFECTED	QUALIFIER

SDG:	REVIEWER:	DATE:	PAGE___OF___
COMMENTS:			

DATA QUALIFICATION SUMMARY - FORM B-7

SDG:	REVIEWER:	DATE:	PAGE___OF___
COMMENTS:			
COMPOUND	QUALIFIER	SAMPLES AFFECTED	REASON

TELEPHONE CONTACT SUMMARY - FORM B-8

REVIEWER:	DATE:	TIME:	PAGE___OF___
COMPANY:			
ADDRESS:			
PERSON CONTACTED:			
TELEPHONE:		FAX:	
PROJECT/CASE/SDG:			
REMARKS:			
ACTION:			

9112 51147

APPENDIX C

EXAMPLE DATA VALIDATION REPORT

DRAFT 7/91

MEMORANDUM

TO:

FROM:

DATE:

SUBJECT: Organic Analysis Data Validation Summary

INTRODUCTION AND SUMMARY

This summary presents the results of data review on Case (case identifier) and SDG (SDG identifier) consisting of twenty (20) low level soil samples submitted for volatile, semivolatile and pesticide/PCB analyses. In addition, this sample set included two (2) equipment blanks and one (1) trip blank. The trip blank was analyzed for all analytical parameters. The samples were analyzed by (laboratory name) using the CLP Statement of Work for Organics (2/88).

DATA QUALIFY OBJECTIVES SUMMARY

The analysis was complete for all requested analyses and met the method and work plan CRQL requirements. Equipment blanks were free of TCL compounds, however, the trip blanks contained low concentrations (<CRQL) of methylene chloride, a common laboratory contaminant. One field duplicate was submitted for all analyses and the results are summarized in Attachment 4.

MAJOR DEFICIENCIES (REJECTED DATA ONLY)

GC/MS tuning criteria were not met for one set of volatile samples (see Attachment 3 - Form B-7) and all volatile data for these samples have been qualified as unusable (R).

All continuing calibration response factor criteria were missed for pentachlorophenol and quantitation limits have been qualified as unusable (R).

MINOR DEFICIENCES (OTHER QUALIFIED DATA)

Semivolatile soils were all extracted between 1 and 2 days out of holding time (from date of collection), and all sample data has been qualified as estimated (J for detects, UJ for non-detects).

Soil volatile surrogate recoveries for the surrogate bromofluorobenzene were missed for 5 samples and all volatile results have been qualified as estimated (J or UJ) (see Attachment 3).

Several TCL compounds were detected in the blanks. Listed below are the maximum concentration of compounds found in all blanks including field blanks. Samples with concentrations of common laboratory contaminants less than ten times (<10X) the highest blank concentrations or less than five times (<5X) for other compounds have been qualified as non-detects in the data summary (see Attachment 3).

Compounds Detected in Blanks	Maximum Concentration (ug/L)
Methylene chloride	12
Acetone	10
Toluene	13
bis(2-Ethylhexyl)phthalate	17
Diacetone alcohol (tentatively identified compound)	1,200

Pesticide/PCB matrix spike and matrix spike duplicate recoveries were exceeded for all soils however DBC surrogates were acceptable and no TCL pesticide/PCB compounds were detected so no data qualification was required.

ATTACHMENTS

ATTACHMENT 1 - Glossary of Data Qualifiers

This attachment provides a glossary explaining all data qualifiers applied as a result of the validation.

ATTACHMENT 2 - As Received Laboratory Sample Concentration Reports

This attachment provides copies of the as received sample concentration reports. This can be provided in a tabular summary similar to that provided in Attachment 4 or can be duplicated copies of the laboratory reports.

ATTACHMENT 3 - Summary of Data Qualifications (Form B-7)

This attachment provides a complete summary of all qualifications applied as a results of the validation.

ATTACHMENT 4 - As Qualified Data Summary

This attachment provides a tabular data summary of all data as qualified from the validation.

ATTACHMENT 5 - Data Review Supporting Documentation

This attachment provides copies of the data validation checklists, data summary forms, telephone contact memoranda and other documentation completed as a results of the data validation.

ATTACHMENT 1

GLOSSARY OF DATA REPORTING QUALIFIERS

U - Indicates the compound or analyte was analyzed for and not detected. The value reported is the sample quantitation limit corrected for sample dilution and moisture content by the laboratory.

UJ - Indicates the compound or analyte was analyzed for and not detected. Due to identified quality control deficiency identified during data validation the value reported may not accurately reflect the sample quantitation limit.

J - Indicates the compound or analyte was analyzed for and detected. The associated value is estimated but the data are usable for decision making processes.

R - Indicates the compound or analyte was analyzed for and due to an identified quality control deficiency the data are unusable.

NJ - Indicates presumptive evidence of a compound at an estimated value.

N - Indicates presumptive evidence of a compound.

ATTACHMENT 2

AS RECEIVED LABORATORY SAMPLE CONCENTRATION REPORTS

(ATTACH DUPLICATE COPIES OF LABORATORY SAMPLE CONCENTRATION REPORTS)

ATTACHMENT 3

SUMMARY OF DATA QUALIFICATIONS

(ATTACH DATA QUALIFICATION SUMMARY-FORM B-7)

DATA QUALIFICATION SUMMARY - FORM B-7

SDG:	REVIEWER:	DATE:	PAGE___OF___
COMMENTS:			
COMPOUND	QUALIFIER	SAMPLES AFFECTED	REASON

ATTACHMENT 4

AS QUALIFIED DATA SUMMARY

(ATTACH TABULATED DATA SUMMARY)

PESTICIDE/PCB ORGANIC ANALYSIS, SOIL MATRIX, (ug/Kg)

Page____ of____

Laboratory																					
Case	SDG																				
Sample Number																					
Location																					
Remarks																					
Sample Date																					
Extraction Date																					
Analysis Date																					
Pesticide/PCB Compound	CRQL	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
alpha-BHC	8.0																				
beta-BHC	8.0																				
delta-BHC	8.0																				
gamma-BHC (Lindane)	8.0																				
Heptchlor	8.0																				
Aldrin	8.0																				
Heptachlor epoxide	8.0																				
Endosulfan I	8.0																				
Dieldrin	16.0																				
4,4'-DDE	16.0																				
Endrin	16.0																				
Endosulfan II	16.0																				
4,4'-DDD	16.0																				
Endosulfan sulfate	16.0																				
4,4'-DDT	16.0																				
Methoxychlor	80.0																				
Endrin Ketone	16.0																				
alpha-Chlordane	80.0																				
gamma-Chlordane	80.0																				
Toxaphene	160.0																				
Arochlor-1016	80.0																				
Arochlor-1221	80.0																				
Arochlor-1232	80.0																				
Arochlor-1242	80.0																				
Arochlor-1248	80.0																				
Arochlor-1254	160.0																				
Arochlor-1260	160.0																				

PESTICIDE/PCB ORGANIC ANALYSIS, WATER MATRIX, (ug/L)

Page____ of____

Laboratory																			
Case	SDG																		
Sample Number																			
Location																			
Remarks																			
Sample Date																			
Extraction Date																			
Analysis Date																			
Pesticide/PCB Compound	CRQL	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
alpha-BHC	0.05																		
beta-BHC	0.05																		
delta-BHC	0.05																		
gamma-BHC (Lindane)	0.05																		
Heptchlor	0.05																		
Aldrin	0.05																		
Heptachlor epoxide	0.05																		
Endosulfan I	0.05																		
Dieldrin	0.10																		
4,4'-DDE	0.10																		
Endrin	0.10																		
Endosulfan II	0.10																		
4,4'-DDD	0.10																		
Endosulfan sulfate	0.10																		
4,4'-DDT	0.10																		
Methoxychlor	0.50																		
Endrin Ketone	0.10																		
alpha-Chlordane	0.50																		
gamma-Chlordane	0.50																		
Toxaphene	1.00																		
Arochlor-1016	0.50																		
Arochlor-1221	0.50																		
Arochlor-1232	0.50																		
Arochlor-1242	0.50																		
Arochlor-1248	0.50																		
Arochlor-1254	1.00																		
Arochlor-1260	1.00																		

SEMIVOLATILE ORGANIC ANALYSIS, SOIL MATRIX, (ug/Kg)

Page____ of____

Laboratory																					
Case	SDG																				
Sample Number																					
Location																					
Remarks																					
Sample Date																					
Extraction Date																					
Analysis Date																					
Semivolatile Compound	CROL	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
Phenol	330																				
bis(2-Chloroethyl)ether	330																				
2-Chlorophenol	330																				
1,3-Dichlorobenzene	330																				
1,4-Dichlorobenzene	330																				
Benzyl Alcohol	330																				
1,2-Dichlorobenzene	330																				
2-Methylphenol	330																				
bis(2-Chloroisopropyl)ether	330																				
4-Methylphenol	330																				
N-Nitroso-di-n-propylamine	330																				
Hexachloroethane	330																				
Nitrobenzene	330																				
Isophorone	330																				
2-Nitrophenol	330																				
2,4-Dimethylphenol	330																				
Benzoic acid	1600																				
bis(2-Chloroethoxy)methane	330																				
2,4-Dichlorophenol	330																				
1,2,4-Trichlorobenzene	330																				
Naphthalene	330																				
4-Chloroaniline	330																				
Hexachlorobutadiene	330																				
4-Chloro-3-methylphenol	330																				
2-Methylnaphthalene	330																				
Hexachlorocyclopentadiene	330																				
2,4,6-Trichlorophenol	330																				
2,4,5-Trichlorophenol	1600																				
2-Chloronaphthalene	330																				
2-Nitroaniline	1600																				
Dimethylphthalate	330																				
Acenaphthylene	330																				
2,6-Dinitrotoluene	330																				
3-Nitroaniline	1600																				

SEMIVOLATILE ORGANIC ANALYSIS, SOIL MATRIX, (ug/Kg)

Page____ of____

Laboratory																					
Case	SDG																				
Sample Number																					
Location																					
Remarks																					
Sample Date																					
Extraction Date																					
Analysis Date																					
Semivolatile Compound	CRQL	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
Acenaphthene	330																				
2,4-Dinitrophenol	1600																				
4-Nitrophenol	1600																				
Dibenzofuran	330																				
2,4-Dinitrotoluene	330																				
Diethylphthalate	330																				
4-Chlorophenyl-phenyl ether	330																				
Fluorene	330																				
4-Nitroaniline	1600																				
4,6-Dinitro-2-methylphenol	1600																				
N-Nitrosodiphenylamine	330																				
4-Bromophenyl-phenylether	330																				
Hexachlorobenzene	330																				
Pentachlorophenol	1600																				
Phenanthrene	330																				
Anthracene	330																				
Di-n-butylphthalate	330																				
Fluoranthene	330																				
Pyrene	330																				
Butylbenzylphthalate	330																				
3,3'-Dichlorobenzidine	660																				
Benz(a)anthracene	330																				
Chrysene	330																				
bis(2-Ethylhexyl)phthalate	330																				
Di-n-octylphthalate	330																				
Benzo(b)fluoranthene	330																				
Benzo(k)fluoranthene	330																				
Benzo(a)pyrene	330																				
Indeno(1,2,3-cd)pyrene	330																				
Dibenz(a,g)anthracene	330																				
Benzo(g,h,i)perylene	330																				

9 1 1 2 5 5 1 4 6 0

SEMIVOLATILE ORGANIC ANALYSIS, SOIL MATRIX, (ug/Kg)

Laboratory																					
Case	SDG																				
Sample Number																					
Location																					
Remarks																					
Sample Date																					
Extraction Date																					
Analysis Date																					
TIC Semivolatile Compound	RT	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q

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SEMIVOLATILE ORGANIC ANALYSIS, WATER MATRIX, (ug/L)

Page____ of____

Laboratory																			
Case	SDG																		
Sample Number																			
Location																			
Remarks																			
Sample Date																			
Extraction Date																			
Analysis Date																			
Semivolatile Compound	CRQL	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
Phenol	10																		
bis(2-Chloroethyl)ether	10																		
2-Chlorophenol	10																		
1,3-Dichlorobenzene	10																		
1,4-Dichlorobenzene	10																		
Benzyl Alcohol	10																		
1,2-Dichlorobenzene	10																		
2-Methylphenol	10																		
bis(2-Chloroisopropyl)ether	10																		
4-Methylphenol	10																		
N-Nitroso-di-n-propylamine	10																		
Hexachloroethane	10																		
Nitrobenzene	10																		
Isophorone	10																		
2-Nitrophenol	10																		
2,4-Dimethylphenol	10																		
Benzoic acid	50																		
bis(2-Chloroethoxy)methane	10																		
2,4-Dichlorophenol	10																		
1,2,4-Trichlorobenzene	10																		
Naphthalene	10																		
4-Chloroaniline	10																		
Hexachlorobutadiene	10																		
4-Chloro-3-methylphenol	10																		
2-Methylnaphthalene	10																		
Hexachlorocyclopentadiene	10																		
2,4,6-Trichlorophenol	10																		
2,4,5-Trichlorophenol	50																		
2-Chloronaphthalene	10																		
2-Nitroaniline	50																		
Dimethylphthalate	10																		
Acenaphthylene	10																		
2,6-Dinitrotoluene	10																		
3-Nitroaniline	50																		

SEMIVOLATILE ORGANIC ANALYSIS, WATER MATRIX, (ug/L)

Page____ of____

Laboratory																							
Case	SDG																						
Sample Number																							
Location																							
Remarks																							
Sample Date																							
Extraction Date																							
Analysis Date																							
Semivolatile Compound	CRQL	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
Acenaphthene	10																						
2,4-Dinitrophenol	50																						
4-Nitrophenol	50																						
Dibenzofuran	10																						
2,4-Dinitrotoluene	10																						
Diethylphthalate	10																						
4-Chlorophenyl-phenyl ethe	10																						
Fluorene	10																						
4-Nitroaniline	50																						
4,6-Dinitro-2-methylphenol	50																						
N-Nitrosodiphenylamine	10																						
4-Bromophenyl-phenylether	10																						
Hexachlorobenzene	10																						
Pentachlorophenol	50																						
Phenanthrene	10																						
Anthracene	10																						
Di-n-butylphthalate	10																						
Fluoranthene	10																						
Pyrene	10																						
Butylbenzylphthalate	10																						
3,3'-Dichlorobenzidine	20																						
Benz(a)anthracene	10																						
Chrysene	10																						
bis(2-Ethylhexyl)phthalate	10																						
Di-n-octylphthalate	10																						
Benzo(b)fluoranthene	10																						
Benzo(k)fluoranthene	10																						
Benzo(a)pyrene	10																						
Indeno(1,2,3-cd)pyrene	10																						
Dibenz(a,g)anthracene	10																						
Benzo(g,h,i)perylene	10																						

SEMIVOLATILE ORGANIC ANALYSIS, WATER MATRIX, (ug/L)

Laboratory																					
Case	SDG																				
Sample Number																					
Location																					
Remarks																					
Sample Date																					
Extraction Date																					
Analysis Date																					
TIC Semivolatile Compound	RT	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q

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VOLATILE ORGANIC ANALYSIS, SOIL MATRIX, (ug/Kg)

Page____ of____

Laboratory																					
Case	SDG																				
Sample Number																					
Location																					
Remarks																					
Sample Date																					
Analysis Date																					
Volatile Organic Compound	CRQL	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
Chloromethane	10																				
Bromomethane	10																				
Vinyl Chloride	10																				
Chloroethane	10																				
Methylene Chloride	5																				
Acetone	10																				
Carbon Disulfide	5																				
1,1-Dichloroethene	5																				
1,1-Dichloroethane	5																				
1,2-Dichloroethene (total)	5																				
Chloroform	5																				
1,2-Dichloroethane	5																				
2-Butanone	10																				
1,1,1-Trichloroethane	5																				
Carbon Tetrachloride	5																				
Vinyl Acetate	10																				
Bromodichloromethane	5																				
1,2-Dichloropropane	5																				
cis-1,3-Dichloropropene	5																				
Trichloroethene	5																				
Dibromochloromethane	5																				
1,1,2-Trichloroethane	5																				
Benzene	5																				
trans-1,3-Dichloropropene	5																				
Bromoform	5																				
4-Methyl-2-pentanone	10																				
2-Hexanone	10																				
Tetrachloroethene	5																				
1,1,2,2-Tetrachloroethane	5																				
Toluene	5																				
Chlorobenzene	5																				
Ethylbenzene	5																				
Styrene	5																				
Xylene (total)	5																				

9 1 1 2 5 1 6 5

VOLATILE ORGANIC ANALYSIS, SOIL MATRIX, (ug/Kg)

Laboratory																					
Case	SDG																				
Sample Number																					
Location																					
Remarks																					
Sample Date																					
Analysis Date																					
TIC Volatile Organic	RT	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q

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VOLATILE ORGANIC ANALYSIS, WATER MATRIX, (ug/L)

Page____ of____

Laboratory																					
Case	SDG																				
Sample Number																					
Location																					
Remarks																					
Sample Date																					
Analysis Date																					
Volatile Organic Compound	CRQL	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
Chloromethane	10																				
Bromomethane	10																				
Vinyl Chloride	10																				
Chloroethane	10																				
Methylene Chloride	5																				
Acetone	10																				
Carbon Disulfide	5																				
1,1-Dichloroethene	5																				
1,1-Dichloroethane	5																				
1,2-Dichloroethene (total)	5																				
Chloroform	5																				
1,2-Dichloroethane	5																				
2-Butanone	10																				
1,1,1-Trichloroethane	5																				
Carbon Tetrachloride	5																				
Vinyl Acetate	10																				
Bromodichloromethane	5																				
1,2-Dichloropropane	5																				
cis-1,3-Dichloropropene	5																				
Trichloroethene	5																				
Dibromochloromethane	5																				
1,1,2-Trichloroethane	5																				
Benzene	5																				
trans-1,3-Dichloropropene	5																				
Bromoform	5																				
4-Methyl-2-pentanone	10																				
2-Hexanone	10																				
Tetrachloroethene	5																				
1,1,2,2-Tetrachloroethane	5																				
Toluene	5																				
Chlorobenzene	5																				
Ethylbenzene	5																				
Styrene	5																				
Xylene (total)	5																				

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VOLATILE ORGANIC ANALYSIS, WATER MATRIX, (ug/L)

Laboratory																					
Case	SDG																				
Sample Number																					
Location																					
Remarks																					
Sample Date																					
Analysis Date																					
TIC Volatile Organic	RT	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q

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ATTACHMENT 5

DATA REVIEW SUPPORTING DOCUMENTATION

(ATTACH COMPLETED DATA VALIDATION CHECKLISTS,
SUMMARY FORMS, AND SUPPORTING DOCUMENTATION)

1100-EM-1 Operable Unit

RADIOCHEMICAL ANALYSES OF GROUND WATER MONITORING WELL SAMPLES

Temp. Well Number	Hanford Well Number	Gross Alpha, pCi/l 1st 2/90	2nd 5/90	3rd 8/90	4th 11/90	5th 2/91
MW-1	S41-E11	8.4	2.0	3.1	0.7	
MW-2	S34-E10	4.4	-0.7	1.8	2.0	
MW-3	S41-E12	17.0	1.7	0.7	3.5	
MW-4	S38-E12A	2.9	ND	4.3	3.8	
MW-5	S38-E12B	3.9	1.8	-2.2	-0.2	
MW-6	S37-E11	3.6	1.9	1.0	-1.8	
MW-7	S38-E11	4.8	0.6	3.3	-1.2	
MW-8	S31-E08	3.8	-3.1	2.2	1.9	
MW-9	S32-E08	1.3	-1.9	0.8	1.4	
MW-10	S30-E10A	11.9	2.2	0.4	4.8	2.0 U
MW-11	S30-E10B	12.2	2.4	6.6	4.2	2.0 U
MW-12	S31-E10A	7.6	4.8	6.7	6.5	2.0 U
MW-13	S31-E10B	9.1	4.1	6.5	5.8	6.4
MW-14	S31-E10C	6.3	4.9	9.6	9.2	3.0 U
MW-15	S31-E10D	9.3	1.6	3.7	5.0	2.0 U
MW-17	S41-E10C	2.2	0.9	0.9	1.6	
MW-18	S37-E12					6.6
	S27-E14		1.6	5.7	3.2	
	S29-E12		1.6	1.1	2.2	
	S30-E15A		-1.4	-1.7	1.5	
	S31-E13		-0.4	-1.2	2.6	
	S32-E13A		-0.5	-0.2	3.3	
	S37-E14	2.2	-1.2	-3.5	-2.4	2.0 U
	S40-E14	1.1	-1.1	-3.4	-1.6	2.0 U
	S41-E13A	ND	0.9	-3.3	-2.7	3.0 U
	S41-E13B	6.0	3.7	-0.7	-1.3	3.5
	S43-E12	2.6	1.9	1.0	0.8	2.0 U
ANF # 14			5.3	22.9		
ANF # 15			37.0	36.7		
ANF # 16			10.0	4.0		
RWF East		ND	-1.0	2.0	-2.3	
RWF West		1.0	-2.0	-0.3	-2.0	2.0 U

ND = Not Detected

Radionuclide Maximum Contaminant Levels (MCL), 40 CFR 141, EPA 1986a:
Gross Alpha . . . . 15 pCi/l

1100-EM-1 Operable Unit

RADIOCHEMICAL ANALYSES OF GROUND WATER MONITORING WELL SAMPLES

Temp. Well Number	Hanford Well Number	Gross Beta, pCi/l				
		1st	2nd	3rd	4th	5TH
		2/90	5/90	8/90	11/90	2/91
MW-1	S41-E11	12.7	3.5	12.1	9.2	
MW-2	S34-E10	8.2	7.3	9.3	11.9	
MW-3	S41-E12	14.7	7.9	12.5	15.0	
MW-4	S38-E12A	7.4	ND	10.6	3.1	
MW-5	S38-E12B	6.5	6.1	6.4	8.9	
MW-6	S37-E11	ND	-1.4	4.1	10.4	
MW-7	S38-E11	6.1	1.4	7.9	9.1	
MW-8	S31-E08	5.3	2.4	9.4	6.1	
MW-9	S32-E08	6.4	1.6	7.6	2.7	
MW-10	S30-E10A	30.2	85.2	5.6	88.9	63.0
MW-11	S30-E10B	35.2	86.5	74.7	81.0	60.0
MW-12	S31-E10A	34.6	87.6	91.0	77.6	61.0
MW-13	S31-E10B	28.8	71.0	81.2	85.8	61.0
MW-14	S31-E10C	35.1	89.4	90.8	89.0	70.0
MW-15	S31-E10D	23.2	51.4	63.6	57.6	46.0
MW-17	S41-E10C	5.6	0.9	2.9	8.1	
MW-18	S37-E12					13.0
	S27-E14		19.7	31.5	14.9	
	S29-E12		1.0	10.5	6.3	
	S30-E15A		2.5	4.7	2.1	
	S31-E13		2.4	7.4	7.3	
	S32-E13A		1.9	11.0	7.9	
	S37-E14	ND	-1.9	1.7	3.9	4.0 U
	S40-E14	ND	-2.5	1.2	0.3	4.0 U
	S41-E13A	0.9	1.3	4.6	5.0	12.0
	S41-E13B	4.9	9.4	11.2	2.8	12.0
	S43-E12	8.8	8.3	10.5	13.8	8.8
ANF # 14			6.5	58.9		
ANF # 15			126.7	98.4		
ANF # 16			58.4	19.1		
RWF East		ND	-2.5	8.1	2.6	
RWF West		ND	-3.6	7.2	4.2	3.0 U

ND = Not Detected

Radionuclide Maximum Contaminant Levels (MCL), 40 CFR 141, EPA 1986a:
Gross Beta . . . . 50 pCi/l

DON'T SAY IT --- *Write It!*

DATE: July 18, 1991

TO: J. A. Lerch T6-08

FROM: S. W. Clark H4-55

Telephone: 6-1513

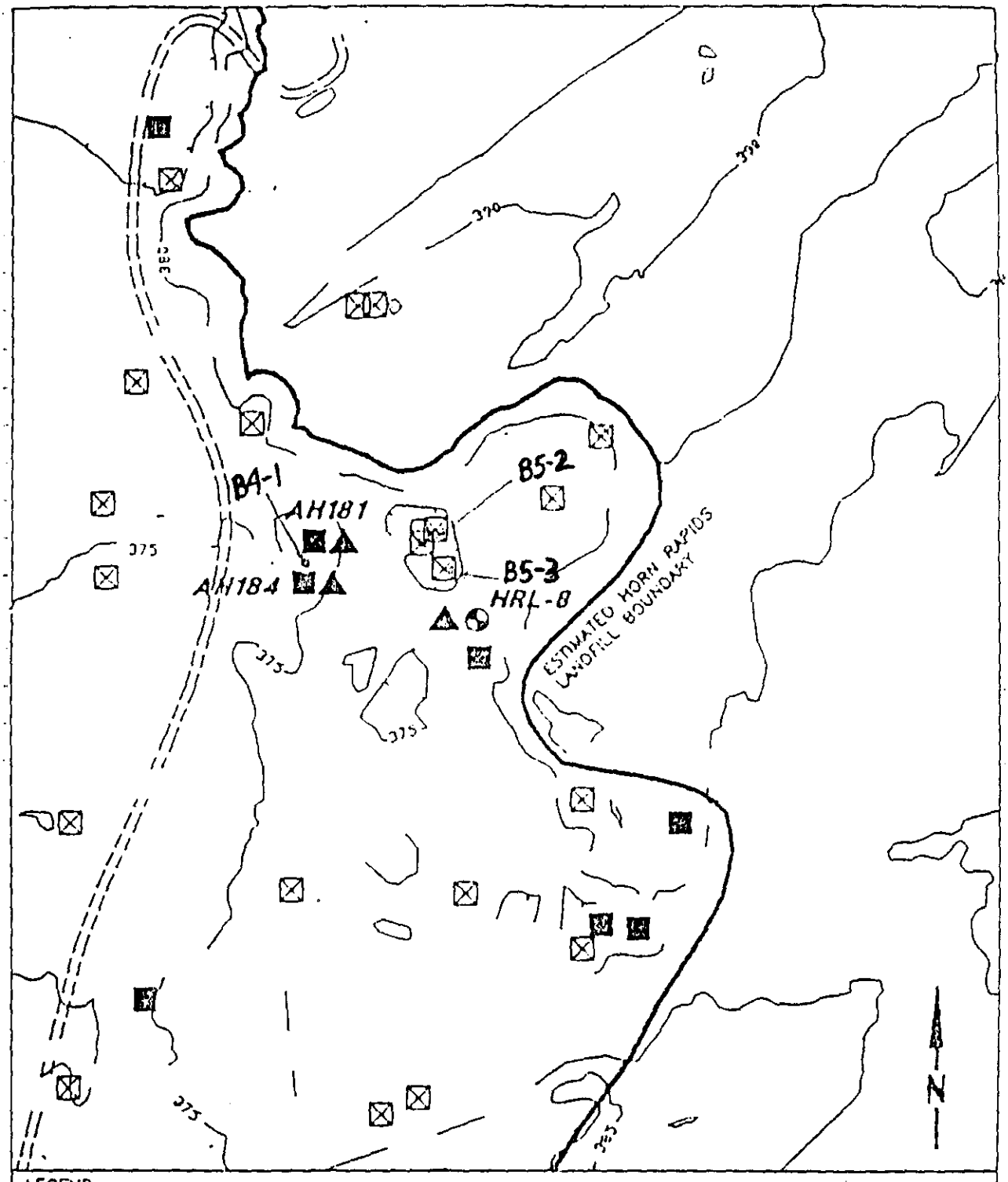
cc: M. J. Lauterbach H4-55
M. R. Adams H4-55
J. H. Kessner T6-08
R. A. Bechtold H4-55

SUBJECT: RADIOCHEMISTRY ANALYSES FROM K-25 LAB

I have tabulated, below, the technetium and thorium assays received from K-25 Lab, with the gross beta assays of the ground water samples for the fourth round of 1100-EM-1 monitoring. As I noted in an earlier cc:mail message, these assays raise more questions than they answer: (1) Is there any significance to the technetium and thorium assays given the high counting errors? (2) Should the activity of the beta emitters, particularly technetium, add up to the gross Beta activity?

Can you arrange to discuss these results with someone familiar with radiochemical assays? Please let me know as soon as possible because Tri-Party Agreement milestones and work to be done by the U.S. Army Corps of Engineers is impacted by lack of knowledge of the species of the gross Beta emitter in the ground water at these wells.

Well No.	Sample No.	Gross Beta, pCi/L	Technetium, pCi/L	Thorium-234, pCi/L
MW-10	B00D29	88.88 +/- 6.6	4220 +/- 1800	-35.8 +/- 33.0
MW-10(D)	B00D33	88.09 +/- 6.6	1280 +/- 1730	(Not Reported)
MW-11	B00D37	80.95 +/- 6.4	1260 +/- 1700	-18.4 +/- 34.0
MW-12	B00B46	77.65 +/- 6.3	5680 +/- 1700	75.50 +/- 39.1
MW-13	B00B51	85.81 +/- 6.5	3350 +/- 1700	24.2 +/- 37.0
MW-14	B00B55	89.02 +/- 6.6	2020 +/- 1700	-34.9 +/- 34.0
MW-15	B00B59	57.65 +/- 5.7	1960 +/- 1700	-31.0 +/- 34.0


B4-1
AH181
AH184
B5-2
B5-3
HRL-8
ESTIMATED HORN RAPIDS LANDFILL BOUNDARY
375
370
365
N
LEGEND:
Phase I RI Surface Soil Sample Location
Phase I RI Surface Soil Sample Location with Chromium above Background
Phase I RI Borehole Location
Proposed Surface Soil Sample Location
Contour interval 5 ft
0
50
100 METERS
0
150
300 FEET

SUMMARY OF ANALYTICAL RESULTS
for
Horn Rapids Landfill B-4 & B-5
(ppm)

Contaminant	B5-3 0-1 ft	B5-3 1-2 ft	B5-2 0-1 ft	B5-2 1-2 ft	B4-1 0-1 ft	B4-1 0-1 ft	B4-1 1-2 ft	Surf. Backgrn (UTL)
Al					14,200	15,800	8,170	7,870
Ba					426	427	206	97.9
Be					1	1.1	0.77	0.65
Ca	5,200	5,050	6,820	5,880	46,600	42,900	18,100	4,530
Cr					12.5	12.9		11.7
Co			15.9	15.4				15.3
Cu	16.7	16.7			31.5	25.3	17.8	16.3
Fe			27,100					27,000
Pb					41	36.3		13.6
Mg					6,380	6,340		5,760
Hg					0.15			
Na	241	277	378	258	4,240	4,450	1,790	112
Zn		54.7	63.2					53.3

Values not reported for contaminants qualified as U or J.
Values not reported for values below back ground uper tolerance limits shown in Phase I RI.

SUMMARY OF ANALYTICAL RESULTS
for
Horn Rapids Landfill B-4 & B-5
(ppb)

Contaminant	B5-3 0-1 ft	B5-3 1-2 ft	B5-2 0-1 ft	B5-2 1-2 ft	B4-1 0-1 ft	B4-1 0-1 ft	B4-1 1-2 ft
2-butanone					35	20	18
toluene					8		9

Values not reported for contaminants qualified as U or J.

TRIP REPORT
MEETING ON GEOPHYSICS RESULTS
HORN RAPIDS LANDFILL, 1100-EM-1 OPERABLE UNIT
JIM MCBANE, CENPW-EN-EE

A meeting was held on 24 July, 1991 at the offices of Golder, Associates, Redmond, Washington. The purpose of this meeting was to clarify the interpretation of the Ground Penetrating Radar (GPR) profiles obtained at the Horn Rapids Landfill (HRL) and to develop recommendations for test pits proposed to be dug at the site for presentation to EPA. Participants in the meeting included:

Bob Anderson, Geophysicist, Golder Associates
Dick Sylwester, Senior Geophysicist, Williamson & Associates, Inc.
Ward Staubitz, Hydrologist, USGS
Joseph Kunk, Senior Scientist, WHC Geophysics Team
Jim McBane, Geotechnical Engineer, CENPW

The initial hour of the meeting was devoted to answering questions regarding the geophysical studies, in general, and the GPR interpretation posed by Mr. Staubitz. A summary of questions and answers are as follows.

1. Why was a threshold level of 300 gammas used in the screening of the data gathered by the magnetometer survey?

The 300 gamma level is widely used as the threshold for similar studies elsewhere. There were no indications at the study site that would indicate a need to modify this value.

2. How do historic GPR surveys performed at sites containing buried drums compare to the data recovered during the current investigation?

Typically, a drum will show as a paraboloid reflection on the data printout. An accumulation of drums should show partial paraboloid reflections on either end of the record. Some reflections recorded during this investigation displayed the paraboloid shape, although no great accumulation of drums appear to be indicated.

3. Soil gas results in the study area are not very conclusive. In a similar investigation for buried drums in the 300 Area, how well did the soil gas monitors indicate the presence of buried drums containing Hexone?

A definite answer to this question was not provided. It was assumed from second hand information, that the soil gas monitors provided a bit more information than in the current case.

4. Does the data from a magnetic survey clearly define buried metallic objects and how does the typical signature appear for an object?

The definition of a buried object will depend on the depth of burial, the nature of the surrounding soil, and the presence of other metallic objects in the vicinity. The typical magnetic signature is a field of high intensity immediately adjacent to one of low intensity, with the object being located at the boundary between the two. However, many factors can alter this ideal signature.

5. During the previously referenced study in the 300 Area, drums were found, but at a depth greater than indicated by the GPR data. The explanation was given that material buried at a shallower depth masked the data to some degree. Is this situation likely to have occurred in this study?

The possibility certainly exists. There is no real method to confirm the geophysical data now in hand without digging into the HRL trenches and comparing observations with the gathered data. Re-interpretation of the data will then be possible.

6. Were any classic barrel reflections, either single or accumulations, found?

Nothing found in the data suggests a classic barrel signature. When all the geophysics are taken as a whole, there is no evidence of other than isolated barrels at the site.

7. How was the figure in the Geophysical Report generated which purports to show locations of buried targets?

The figure was prepared using GPR printouts which show intensity of reflections through the use of color. The targets were selected from color, processed data. The data is actually recorded by GPR instruments as millivolts received by the antenna. Better reflectors provide a higher energy return and thus the record shows a higher intensity. This is qualified by the fact that the instruments are only really capable of recording the contrast between objects in the subsurface. Thus a highly reflective object buried in a very reflective soil will not have the same intensity of signature as the same object in an non-reflective soil.

8. How were the 11 recommended locations for test pits selected?

Targets in the recommended pit locations had the highest intensity readings of data gathered during the geophysical survey. The shape of the GRP returns suggested a barrel-like signature. The magnetics supported the interpretation of metallic reflectors. However, nothing can be stated with certainty until pits are excavated and the records can be

compared to actual materials found in the subsurface.

9. How will the selection of test pit locations and depths affect the potential dispute concerning the contaminant plume which is apparently emanating from the Advanced Nuclear Fuels (ANF) complex?

Unless all pits are excavated to confirm geophysical data gathered at HRL, there will always be an opening for ANF to claim insufficient characterization was performed at the site. At least some test pits will be needed to support the findings of the geophysics survey.

After all questions had been addressed, the meeting focus shifted to the actual GPR records. Mr. Anderson explained the reasoning behind the selection of each recommended test pit, supported the choices by identifying evidence visible on the actual records, and answered any questions generated as a result of his explanations. The participants made recommendations to Mr. Staubitz as to the test pit geometry and priority; to be relayed to regulators responsible for the final decision on the test pit program.

Test Pit #1: The GPR record indicated a fairly shallow target (3-4 ft. depth) which presented a paraboloid shape. The recommendation of a 12-foot deep test pit was agreed upon with no discussion to the priority.

Test Pit #2: The target which was used to select this site is located at a depth of 3-4 feet. A strong ringing was recorded in the GPR record and magnetics indicated a high anomaly. Other parabolic signatures apparent in the area were not consistent with adjacent GPR survey lines nor did they correspond to magnetic anomalies. The consensus for this test pit was a 12-foot depth and a high priority.

Test Pit #3: Two paraboloid targets are indicated by the GPR record; one shallow (3-4 feet), one deep (15-20 feet), and close together in a lateral sense. The shallow target would likely be intercepted by a pit intended to reach the lower target. The recommendation of placing a secondary priority on pit 3 was agreed upon with the depth issue to be left to the regulators.

Test Pit #4: The target at this location is shallow (3-4 feet). The magnetic signature was not large, but it was significant. A 12-foot deep pit having a high priority was recommended. A permanent soil gas probe is located in close proximity to this site. Experts will have to be consulted as to the potential for an excavation

Test Pit #5: There is an "eye-catching" parabolic signature in the vicinity of the soil gas probe. It appears to be a

shallow, single target rather than an accumulation of targets. It is a good candidate to use as a diagnostic for further interpretation of the GPR records. A secondary priority was assigned by the group, with an indefinite depth of less than 12 feet.

Test Pit # 6: The target is located at an intermediate depth of approximately 6-feet. The record depicts numerous targets within a large area which lack any significant magnetic anomaly. A depth of 12-feet was recommended with no real priority recommendation. A north-south oriented trench of 30-foot length may be an alternative to a single pit.

Test Pit #7: Pit 7 is the only recommended intrusive investigation recommended for that particular waste trench. The target used to select this site is shallow and fairly isolated; however, the entire record is chaotic. A 12-foot deep pit was recommended having a lower priority rating. The regulators will need to make a decision on this location as it is considered a low priority but it is the only pit planned for this particular trench.

Test Pit #8: The GPR data indicated a broad, shallow target having a gross parabolic shape. There is a magnetic anomaly which may correspond to the GPR reflection. This is considered a low priority site having a low priority. This site may be outside of the asbestos filled trench. But there is a possibility that asbestos may be present.

Test Pit #9: This pit is located within the signed asbestos pit area. Selection of this site was based predominantly on magnetics due to the chaotic record gathered with the GPR. Some definite targets can be found on the GPR record, but this record presents a different character to other records of the HRL. The data is more "uniformly chaotic". Additional GPR lines are recommended for this site prior to digging. The current data does not meet the modelling criteria for buried drums. It is recommended that this site be given a low priority so the asbestos trench not be opened unnecessarily. A final recommendation will be offered after other test pits are completed and the data evaluated.

Test Pit #10: The character of data and the recommendations of meeting participants are identical to pit 9.

Test Pit #11: Records were not available for inspection for pit 11. This investigation is located in study area B, north of the main landfill body, in the vicinity of the metal burning cage. A well defined, shallow target was identified during data analysis. This pit was assigned a high priority and a 12-foot depth for presentation to the regulators.

The meeting content was summarized prior to adjourning. The following items were deemed significant.

1. Digging within the asbestos trench was not recommended at this time. Other test pits should be first completed, and the GPR data re-analyzed based on the excavation findings. \

2. The geophysics specialists present at the meeting agreed that the gathered data does not support anecdotal information of large numbers of barrels buried in the landfill.

3. None of the currently identified anomalies approach the intensities found during previous investigations where buried barrels were found.

The meeting adjourned at approximately 1 PM.

James A. McBane
25 July 1991

APPENDIX B

PROJECT _____ FIELD TEAM LEADER _____

SAFETY, TRAINING AND PERSONNEL REQUIREMENTS FOR HAZARDOUS WASTE SITE WORKERS

1. NAME: _____
   JOB TITLE: _____
   PAYROLL NUMBER: _____
   SOCIAL SECURITY NUMBER: _____
   COMPANY: _____

2. THIS WORKER IS A PARTICIPANT IN THE COMPANY MEDICAL SURVEILLANCE PROGRA
   AS REQUIRED BY OSHA 29 CFR 1910.120 AND IS MEDICALLY CLEARED TO PERFORM
   WORK AT A HAZARDOUS WASTE SITE.

   YES_____ NO_____

   ANY MEDICAL RESTRICTIONS? YES_____ NO_____

3. SAFETY EQUIPMENT:

	REQUIRED YES	NO
HARD HAT	___	___
SAFETY GLASSES	___	___
SUBSTANTIAL FOOTWEAR	___	___

4. TRAINING:

	DATE COMPLETED
40-HOUR HAZARDOUS WASTE WORKER (WHC 020201), OR 8-HOUR ANNUAL REFRESHER (WHC 020060) OR EQUIVALENT	_____
24-HOUR WASTE SITE FIELD EXPERIENCE (WHC 020202) OR EQUIVALENT	_____
MASK FIT FROM HEHF	_____
RADIATION WORKER INITIAL TRAINING (WHC 020001) OR RAD WORKER REQUALIFICATION TRAINING (WHC 020003)	_____
SCOTT SKA PAK MSA PAPR (WHC 020032)	_____

5. ADDITIONAL TRAINING:	REQUIRED YES	REQUIRED NO	DATE COMPLETED
SELF-CONTAINED BREATHING APPARATUS (SCBA- WIIC 0200030)	___	___	_____
OSHA 8-HOUR SUPERVISOR TRAINING (WIIC 020250) OR EQUIVALENT	___	___	_____
FIRST AID (WIIC 020055)	___	___	_____
NOISE CONTROL (WIIC 020194)	___	___	_____
OTHER:_____	___	___	_____
OTHER:_____	___	___	_____

_____ IS MEDICALLY CLEARED TO PERFORM HAZARDOUS WASTE WORK. THE REQUIRED TRAINING HAS BEEN VERIFIED AS COMPLETE AND THE ABOVE EMPLOYEE IS QUALIFIED TO WORK ON A HAZARDOUS WASTE SITE.

IF THERE ARE ANY CHANGES THAT AFFECT THE STATUS OF EITHER THE MEDICAL CLEARANCE OR THE TRAINING CERTIFICATION DURING THE DURATION OF THE WORK (SUCH AS MEDICAL RESTRICTIONS OR EXPIRED TRAINING), THE ENVIRONMENTAL HEALTH AND PESTICIDES SERVICES MANAGER WILL BE NOTIFIED IMMEDIATELY.

MANAGER:_____

ORGANIZATION:_____

DATE:_____

DON'T SAY IT --- Write It! **DATE:** August 1, 1991

TO: Ms E. A. Bracken
DOE/RL- ERD

FROM: Wendell Greenwald USACE

Telephone: 6-9698

cc:

Steve Clark	WHC
M. J. Lauterbach	WHC
M. R. Adams	WHC
T. M. Wintezak	WHC
R. K. Stewart	DOE/RL ERD
J. T. Stewart	USACE

SUBJECT:
Incident Report - Unauthorized Disturbance of the Ephemeral Pool

As requested by your office, a report on the unauthorized disturbance of the Ephemeral Pool, 1100-EM-1 Operable Subunit is provided.

On July 29, 1991, a roadway maintenance crew graded the parking lot shoulder area adjacent to the Ephemeral Pool (see Attachment 1 for area graded). The grading operation consisted of scarifying the soil to a two to three inch depth and working this loosened soil to provide a smooth surface. Water was applied to effectively control dust. The work in the area adjacent to the Ephemeral pool was approximately 2-1/2 hours in duration. The grading activity did not encroach into any area containing PCB's above quantitation limits. Some slight encroachment may, or may not have occurred into areas of the Ephemeral pool contaminated by low levels of the pesticide chlordane. Neither the individuals operating the equipment nor the Fleet Maintenance Managers supervising them were aware that the Ephemeral Pool area was a past practice waste site. Additionally, no signs were posted to indicate that the area was under investigation as a past practice waste site.

In response to this near intrusion into a past practice waste site, the following actions have been taken:

- a meeting of managers and environmental specialists was quickly convened to determine the health risks posed to the work crew, the appropriate measures required to control access to the 1100-EM-1 Operable Subunits, the regulations and directives which may have been violated, the reports required to document the incident occurrence, and the appropriate changes required in existing procedures;

- the health hazard posed by exposure of the work crew to the maximum PCB concentrations present at the Ephemeral Pool was computed (a very conservative assessment considering the crew did not enter the PCB contaminated portion of the Ephemeral Pool) to be 4.34×10^{-9} which is inconsequential considering the EPA point of departure is 10^{-6} (see Attachment 2 for details of the hazard computation);

- a medical examination (at HEHF) of the grader operator will be provided (if the operator agrees), even though the crew did not enter the PCB area and inhalation of contaminated dust was eliminated by dust control procedures; and

- signs have been placed around the Ephemeral Pool (on July 31, 1991) and are in the process of being installed around the remaining 1100-EM-1 Operable Subunits (see Attachment 3 for description of signing around each Operable Subunit).

Several actions are proposed which will aid in preventing an incident similar to the disturbance of the Ephemeral Pool. An Internal Memo requesting changes in the procedures will be issued and is shown in Attachment 4. The following changes are being recommended:

- the excavation permit process should be made more comprehensive so that grading of waste sites and other disturbance activities would be covered under a permit process;

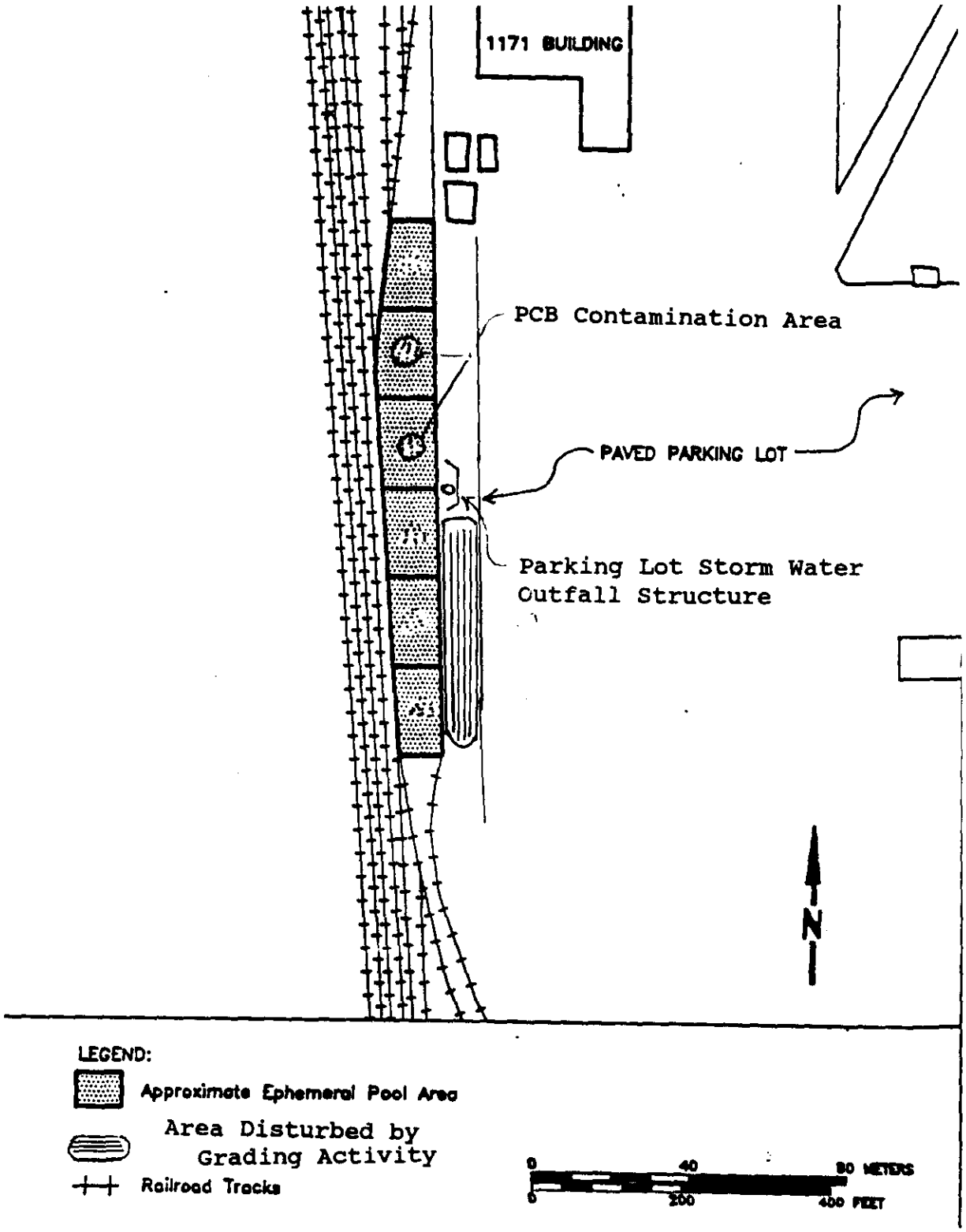
- the Environmental Compliance Manual (WHC-CM-7-5, Parts L and M) should be revised to adequately cover mixed waste sites;

- the facility managers/landlords should be educated on the pertinent portions of the Environmental Compliance Manual; and

- all Hanford personnel should be educated (via HGET) on access limitations and procedures for inactive waste sites.

Additionally, the facility managers/landlords should be included in the information loop on activities occurring at past practice waste sites within their area of responsibility. This will be accomplished for the 1100-EM-1 by including the facility manager/landlord in the distribution of Unit Manager Meeting minutes.

In summary, the unauthorized disturbance at the Ephemeral Pool did not encroach significantly into the contaminated area and no health risks are posed by this incident Posting of the 1100-EM-1 Operable Subunits has begun so that a similar incident will not reoccur. Changes to procedures have been proposed which will reduce the likelihood of a similar problem occurring at other past practice sites at Hanford.



Attachment 1

health risk and is not the focus of any current investigation. Controlling access to the site, even with such a minor control as installing signs, will interfere with maintenance operations in that area. Therefore, installing signs at this site is neither warranted nor feasible.

UN-1100-6, DISCOLORED SOIL SITE: Access to this site is limited by the irrigation canal to the north and a sand dune to the south. One sign located at the north east end of the site will effectively control access to the site.

SOUTH PIT: The waste site identified as the South Pit is located on property owned by Advanced Nuclear Fuels Corp. (ANF). Posting this site will be recommended to ANF during review of their remedial investigation work plan in August 1991.

Westinghouse Hanford Company

Internal Memo

From: Environmental Engineering Group 81220-91-141
Phone: 6-8361 H4-55
Date: July 31, 1991
Subject: IMPROVED ACCESS CONTROL TO INACTIVE WASTE SITES

To: R. E. Lerch B2-35

cc: L. C. Brown H4-51
G. D. Carpenter B2-16
C. J. Geier B2-19
T. M. Wintczak L4-92
MRA: File/LB

Reference: Letter, D. R. Einan (EPA) to R. K. Stewart (RL), "Potential Unauthorized Access to Hazardous Waste Investigation Areas," 9102888, dated July 15, 1991.

As you requested, guidance is provided to improve access control to inactive waste sites in response to the letter from D. R. Einan to R. K. Stewart (see reference). The need for improved access control sitewide is exemplified by the grading incident that occurred at the 1100-EM-1 "ephemeral pool/Hanford Pond" site the week of July 29, 1991. Although corrective action has been taken specifically at 1100-EM-1, similar incidents have happened in the past and will continue to happen until the following recommendations are enacted:

- Implement the recommendations directed to G. D. Carpenter on November 16, 1990, (see attached letter). These recommendations are still valid given the recent 1100-EM-1 incident.

- Revise the Environmental Compliance Manual, WHC-CM-7-5 Parts L and M to adequately cover mixed radioactive/hazardous waste sites. The focus of many of the provisions in this part are still exclusively related to only radioactive contaminants/sites. Numerous procedural gaps and inconsistencies regarding inactive waste sites exist in the manual.

- Train all facility managers/landlords on the provisions of WHC-CM-7-5 relating to inactive waste sites. In the recent 1100-EM-1 incident, the facility manager/landlord was not aware of the existence of WHC-CM-7-5 even though numerous responsibilities for facility managers/landlords are listed in the document.

- Train all Hanford personnel via HGET on access limitations and procedures for inactive waste sites.

Attachment 4

The recommended actions above are listed in the order they should occur. If you have any questions please contact me on 6-8361.

M R Adams

M. R. Adams
Manager

tle

Attachments

Westinghouse Hanford Company

Internal Memo

From: Environmental Engineering Group 81220-90-396
Phone: 6-8361 H4-55
Date: November 16, 1990
Subject: RECOMMENDATIONS FOR IMPROVED CONTROL AND COORDINATION OF ACTION IN OPERABLE UNITS

To: G. D. Carpenter B2-16

cc: L. C. Brown H4-51
Distribution
MRA File/LB

Recently interface meetings have been held as required by a DOE-RL audit to improve communications between site planning, projects, and remedial investigation personnel. The meetings are held to resolve potential problems related to construction in or near waste sites and the potential impacts of new facilities on planned remedial investigations. As a result of the interface meetings, a number of problems and solutions have been identified regarding control of activities within operable units (OU). The solutions recommended will not result in additional procedures or approvals, but will enhance the ability to implement the new Hanford vision set forth by senior management.

These problem areas identified include:

- Landlordship responsibility is not being taken by any organization for many areas and sites within an OU. This results in the Environmental Engineering, Technology and Permitting Function (EET&PF) preparing unusual occurrence reports for contamination zones discovered in an OU simply because a well is being drilled in the OU.

- The current excavation permit process is not comprehensive enough in that items such as, removal of contaminated plants or construction of barriers to prevent animals into contaminated areas are not covered within the permit process. The result has recently been regulatory agency concern about actions taken onsite to remove contaminated mulberry bushes and to prevent birds intruding into contaminated sediments. A more comprehensive excavation permit process would have provided greater internal awareness and approval of these types of actions so that the regulatory concerns could be more effectively addressed with the Regulators.

Attachment

To address these problem areas, the following corrective measures are recommended:

- The procedure regarding excavation permits (WHC-CM-8-7, 503.1) should be renamed and revised to cover a broader range of site disturbances, including placing of fill on waste sites, removal of vegetation, etc. It is suggested that excavation permits be renamed "disturbance" permits. This suggestion will require a change in WHC-CM-8-7, 503.1. A form to accomplish this change is attached for your convenience.

- All employees should be informed of the need to obtain a "disturbance permit" (excavation permit) for certain activities. A name and number should be provided to all employees to obtain information on how to obtain the permit. Employees could be informed by a handbill sent to all employees with a number to call if certain activities are to be conducted (see attached example handbill). Basically the employee would call a "call disturb" number if certain actions were planned. This service should be handled by Environmental Protection.

- Before signing the "disturbance permit" (excavation permit), Environmental Protection (Environmental Assurance) should complete a checklist of notifications completed within the Environmental Division including the relevant OU technical coordinator within the EET&PF, the RCRA site cognizant engineer within Regulator Permitting, and other relevant organizations within the Environmental Division. This would not increase signatures required, but would provide for adequate notification prior to approval. The contact person within Environmental Protection will be provided with up-to-date OU maps, status of on-going remedial investigations, names of OU technical coordinators, etc.

- The Cultural Resources Review form should be modified slightly with distribution to the same office within Environmental Protection that will handle the new "disturbance permit." The coordinator would send these forms to the same distribution (per checklist) that is notified on disturbance permits. This provides a first notification of field disturbance activities in advance of the disturbance permit. The disturbance permit becomes the "second trap" for notification of the activity.

- MRP 5.10, "Solid Waste Management Units/Operable Unit Management," will be completed. This procedure refers to the Waste Information Data System (WIDS) which designates a landlord (Solid Waste Management Unit Manager) for each site and area within an OU. Of particular concern is the designation for outdoor sites in inactive OUs that are not within facility boundaries and currently have no landlord. The EET&PF should not be a landlord since it is not an operations type group. It is recommended that Restoration Operation be given landlordship of these sites.

- In able to support the above recommendations, an interactive (readily update) map system needs to be established. The current H-2 drawing system is not deemed responsive enough to enable updates on an almost daily basis.

Since the recommendations above are related to Environmental Protection and Assurance, they are directed to you for action. If I can be of assistance, please contact me at 6-8361.

M R Adams

M. R. Adams, Manager
Environmental Engineering Group

jew

Westinghouse Hanford Company	CONTROLLED MANUAL DOCUMENT CHANGE REQUEST (CMDCR)	
1 Manual Number	4. Manual Title	6. CMDCR Number
2. Section Number / 3. REV	5. Section Title	7 Date

8. Change Request

- ☐ Reflects changes to requirement or interfacing documents, etc.
- ☐ Responds to deficiency documents (identify commitment dates in block 10)
- ☐ Other

9. Initiator _____

Organization _____

MSIN _____ Phone _____

10 Management Standards Point-of-Contact

_____

MSIN _____ Phone _____

11 Proposed Change

12. Responsible Organization Disposition

☐ Immediate incorporation required ☐ Will be included in subsequent revision ☐ Disapproved (state reason below)

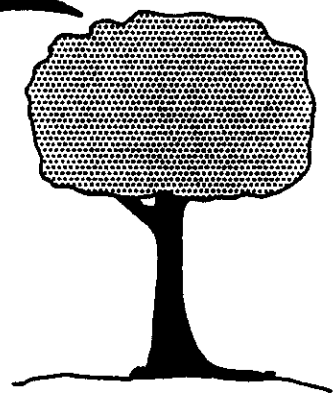
13. Manager – Responsible Organization, or CM Point-of-Contact	Organization	Date

A-6000-269 (03/89)

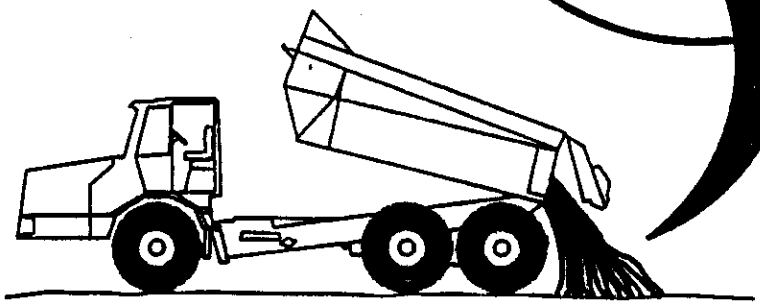
BEFORE YOU DO THE FOLLOWING:

DIG

DIAL "9-DISTURB"



REMOVE VEGETATION



PLACE FILL

DISTRIBUTION 81220-90-396

R. C.	Brunke	H4-57
K. S.	Daly	B4-64
J. M.	Frain	H4-55
R. R.	Gadd	B3-65
J. M.	Garcia	R3-12
C. J.	Geier	H4-57
V. Q.	Hale	H4-15
W. L.	Johnson	H4-55
D. B.	Jordan	B4-64
D. S.	Kelly	B3-65
M. J.	Lauterbach	H4-55
R. E.	Lerch	B2-35
R. A.	Lujan	N1-22
E. G.	See	H4-22
S. W.	Seiler	B4-64
L. C.	Vanselow	R3-27
T. C.	Varljen	B3-65
A. K.	Vogt	N1-30
S. W.	Wiegman	B2-19

Change Number	FEDERAL FACILITY AGREEMENT AND CONSENT ORDER CHANGE CONTROL FORM	Date
M-15-91-2	Do not use blue ink. Type, or print using black ink.	8/11/91

Originator: John T. Stewart

Phone: 376-9101

Class of Change

☐ I – Signatories (Section 13.0) ☒ II – Project Manager ☐ III – Unit Manager

Change Title

REVISION TO MILESTONES M-15-01B AND M-15-01C

DRAFT

Description/Justification of Change

Change Interim Milestone M-15-01B due date from Nov. 1991 to Jan. 1993.

Change Interim Milestone M-15-01C due date from Apr. 1992 to Jan. 1993.

Consolidate Interim Milestones M-15-01B and M-15-01C into Interim Milestone M-15-01B/C.

(See Page 2 for Justification of Change)

Impact of Change

Deferral of Interim Milestones M-15-01B and M-15-01C.

Affected Documents

The Hanford Federal Facility Agreement and Consent Order, Volume 2 dated March 1990, Appendix D, Table D-2 and Figure D-1.

Approvals ___ Approved ___ Disapproved

DOE _____ Date _____

EPA _____ Date _____

Ecology _____ Date _____

Description and Justification of Change

The change in schedule for TPA milestones M-15-01B and M-15-01C is requested to allow identified Remedial Investigation/ Feasibility Studies (RI/FS) activities to be accomplished and incorporated into a consolidated Final RI/FS Report for the 1100-EM-1 Operable Unit. Attachment 1 is a revised schedule outlining the activities to be accomplished and a submittal milestone (M-15-01B/C) for the Final RI/FS Report of December 1992.

Change Number M-15-91-1, Revision to Milestones M-15-01B and M-15-01C, was submitted June 20, 1991 and denied by EPA June 27, 1991 and by Ecology July 1, 1991. DOE-RL raised the issue to Formal Dispute in accordance with procedures outlined in the TPA. The Unit Managers met several times during the informal dispute resolution phase to discuss the dispute and attempt to reach resolution. These meetings resulted in agreement on the scope of RI/FS activities remaining to complete this project, and approximate durations for each. Attachment 2 is the meeting minutes and list of agreements.

EPA and Ecology Project Managers agreed with and supported their respective Unit Managers, but questioned whether DOE-RL had "Good Cause" for extending the TPA Milestones. Attachment 3 is a copy of the letter dated July 26, 1991 from EPA and Ecology Project Managers approving the Scope of remaining activities and the time durations associated with each, and presenting their concerns for approving a schedule extension.

Attachment 4 is the Dispute Statement, with submission letter, presenting the justified good cause arguments for the requested time extension.